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NONMETALS TEST AND EVALUATION
Delivery Order 0006: Support for Aging Aircraft Technology Transition

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THIS TECHNICAL REPORT IS APPROVED FOR PUBLICATION.

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14. ABSTRACT
The purpose of this effort was to assist the transition of several technologies developed by AFRL/MLSA to Air Force maintenance units. Work on three specific technologies was conducted: (1) sol-gel prebond surface preparations, (2) development of a composite materials database for the design of composite patches and (3) documentation of material and processing repair procedures. The bulk of the effort for this program was on Tasks 1 and 2. Very little work was requested by AFRL/MLSA for Task 3. Sol-gel prebond surface preparations are of interest to Air Force maintenance organizations in order to provide easier and safer prebond surface preparations for aluminum structure as compared to currently used processes. UDRI evaluated materials, processes, and refined processing windows to ease transition of the sol-gel surface preparations to maintenance organizations across the Air Force. The sol-gel work consisted of a series of smaller projects aimed at specific topics of interest from maintenance organizations. Work on the composite materials database development centered on determining vacuum cure cycles for commonly used graphite/epoxy prepreg systems, performing mechanical testing on lower-temperature, vacuum-cured composite materials and calculating B-basis design allowables for these materials when processed under on-aircraft repair conditions.

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PREFACE

This report covers work performed by the University of Dayton Research Institute (UDRI) from 10 January 2003 until 30 September 2005. This work was performed on Contract F33615-00-D-5600, Delivery Order 0006, through AFRL/MLSA. The program was funded through the U. S. Air Force Aging Aircraft Systems Squadron as part of the Bonded Repair Capability Enhancements Advanced Technology Demonstrator (ATD) Program and through the Environmental Security Technology Certification Program (ESTCP) under Project PP-0204. This Delivery Order was administered under the direction of the Materials Integrity Branch of the Systems Support Division, Air Force Materials and Manufacturing Directorate, Air Force Research Laboratory, Wright-Patterson Air Force Base, Ohio. Mr. James J. Mazza was the contract monitor and provided technical expertise throughout the program. The UDRI Contract Program Manager was Mr. Roger Rondeau. The UDRI Technical Lead was Mr. Dan McCray. Technical support was provided by Mr. Jeffrey Smith (UDRI) and Mr. Paul Childers (UDRI). Additional technical support was provided by Mr. Justin Rausch, Ms. Kristen Shiverdecker, and Ms. Kelly Feirstine from the Southwestern Ohio Council for Higher Education (SOCHE). Administrative support was provided by Ms. Jeanne Miller, UDRI.

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1 INTRODUCTION

The purpose of the work performed under this delivery order was to enhance the ability for aircraft maintainers to design and perform bonded repairs. In order to successfully accomplish this, three separate tasks were provided by AFRL/MLSA for UDRI to investigate:

1. evaluation of prebond metal surface preparation,
2. generation and documentation of bonded repair materials property data, and
3. documentation/dissemination of bonded repair materials property data.

Efforts for each of these tasks are described in separate sections of this report. The prebond surface preparation test is documented in detail. Only a brief overview is provided for the materials property data task, with a reference to a separate limited distribution report for details. Very little was accomplished under the third task since the anticipated workload from AFRL/MLSA was not provided.

2 TASK #1: EVALUATION OF PREBOND METAL SURFACE PREPARATIONS

High-performance surface preparations for adhesive bonding of metals typically require the use of strong acids or bases, volatile organic compounds (VOCs), and hexavalent chromium. Surface preparations used for on-aircraft repair of aluminum typically rely on hazardous materials or inconvenient processing steps, or they do not yield adequate bond performance. Grit-blast/silane (GBS)[1], phosphoric acid containment system (PACS)[2], which is a version of phosphoric acid anodize (PAA)[3], and certain acid paste etches are the high-performance surface treatments currently available for on-aircraft application. All are used in conjunction with chromated, high-VOC primers. All are time consuming for on-aircraft repair. Furthermore, their use is becoming more difficult due to existing and proposed environmental, safety, and health regulations.

Previous work by AFRL/MLSA and the University of Dayton Research Institute (UDRI) optimized processes using sol-gel coatings for aluminum prebond surface preparation in order to reduce the environmental impact of the above-mentioned hazardous materials[4]. The effort involved development of processes based on The Boeing Company's Boegel-EPII[5] sol-gel chemistry. The purpose of Task #1 under this contract was to perform additional materials screening, process development, and testing in order to transition the sol-gel surface preparations to Air Force maintenance organizations.

Several projects were identified and evaluated under this task. Each of those projects has been detailed separately in the following sections.

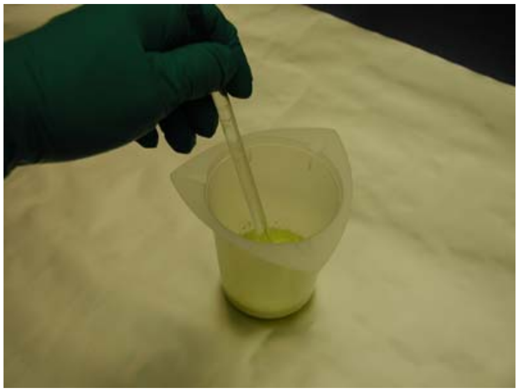
2.1 BRUSH APPLICATION METHOD FOR BR 6747-1

Adhesive bonding of metal substrates typically requires the use of adhesive bond primers for optimum bond durability and to protect prepared surfaces prior to adhesive bonding[6]. Adhesive bond primers are designed to be applied in original equipment manufacturing (OEM) environments via spraying. However, spray-applying adhesive bond primers can be difficult and impractical, particularly for on-aircraft repair or other times when components cannot be sprayed in a controlled environment. Therefore, nonspray application methods are often the method of choice for aircraft maintenance personnel. Past efforts to develop a nonspray technique for

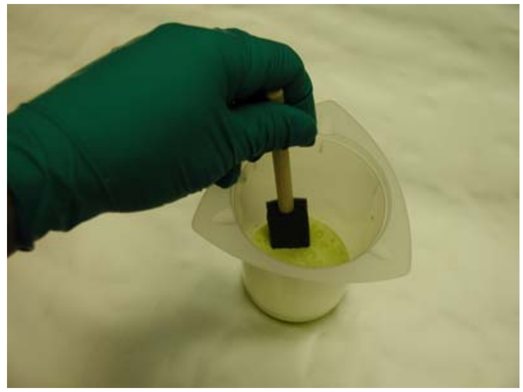
Cytec Engineered Materials (Cytec) BR 6747-1 waterborne bond primer with sol-gel surface preparations were not fully successful since adequate failure modes were not always achieved in laboratory testing[7]. Therefore, a project was initiated to develop a practical, effective, nonspray application technique for BR 6747-1 using commercially available foam paintbrushes.

2.1.1 Brush Application Technique

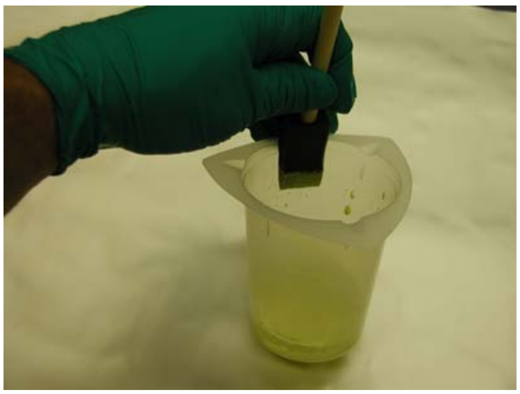
The evaluated brush technique for Cytec BR 6747-1 (20% solids) waterborne primer was performed on Al 2024-T3 adherends that were treated using phosphoric acid anodize (PAA)[3] process so the primer thicknesses could be readily measured using an eddy current technique (Isoscope from Fisher Scientific). Use of the Isoscope for measuring primer thickness on grit-blasted or nylon pad-abraded surfaces is difficult to perform since the instrument is sensitive to surface roughness. Cytec recommends applying BR 6747-1 to a thickness between 0.1-0.4 mil (0.0001-0.0004 inch). The most promising brush technique, illustrated in Figure 1, uses common foam paintbrushes available at many home improvement/hardware stores, such as Lowe's and Home Depot. Several sets of Al 2024-T3 color chips were fabricated to estimate the primer thickness for grit-blast and nylon pad-abraded test specimens. Paint chips were fabricated, as shown in Figure 2, so grit-blasted and nylon pad abraded areas were located between two areas of PAA. Once the entire chips were primed, the primer thickness of the two PAA ends was accurately measured with the Isoscope. The primer thickness in the center of the panel was then interpolated between the two PAA ends.



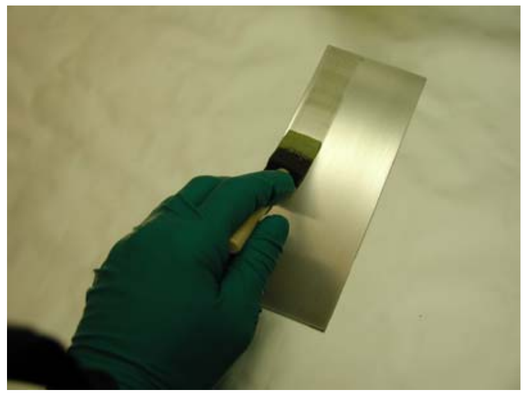
1) Thoroughly Mix BR 6747-1 Primer



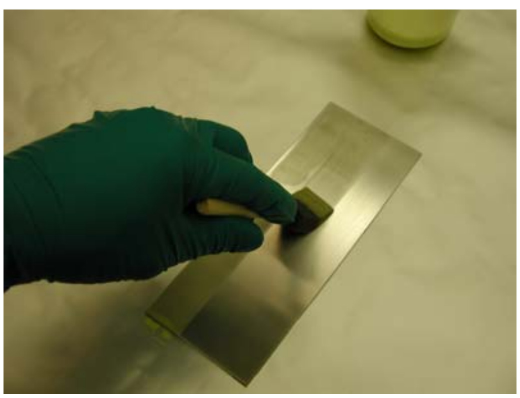
2) Saturate foam brush with primer



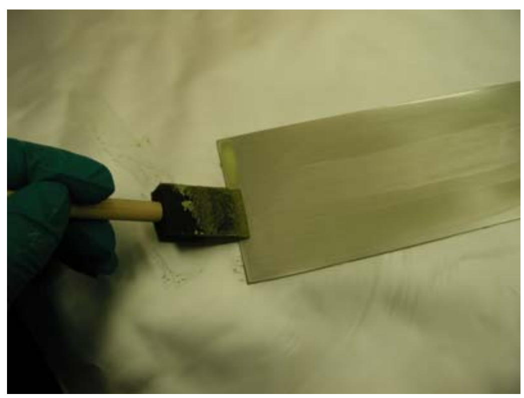
3) Dab excess primer from brush



4) Apply Primer in a Single Pass



5) Slightly overlap for full coverage



6) Blot excess primer from edge

Figure 1: Brush Application Process Description

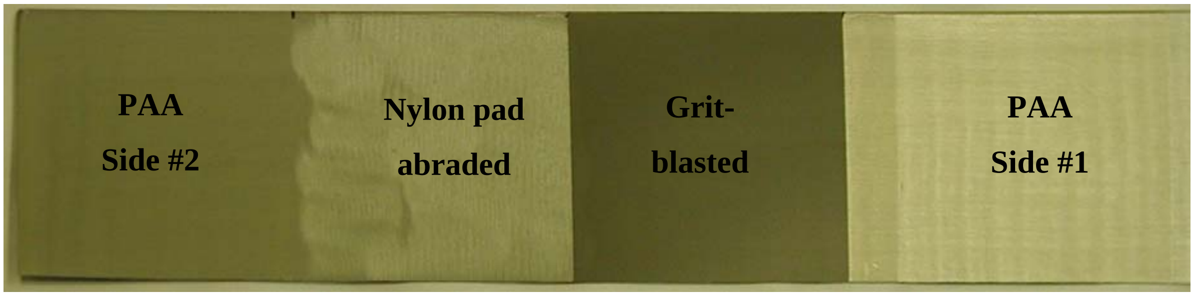


Figure 2: BR 6747-1 Color Chip #3

2.1.2 Mechanical Testing Materials and Processes

Al 2024-T3 adherends were used for all testing. Three different surface preparations were evaluated in this effort: PAA, grit-blast/sol-gel[8], and nylon-pad/sol-gel[9]. A brief description of the sol-gel surface preparations is provided in Table 1. AC-130 sol-gel solution (commercially available from Advanced Chemistry & Technology, Inc. in Garden Grove, CA) was mixed according to the manufacturer's recommendations for each sol-gel surface preparation. Cytec BR 6747-1 primer (20% solids) was applied to a nominal thickness of 0.2 mil (0.0002 inch) using the foam paintbrushes. The primer was dried at ambient laboratory conditions (60°F-70°F and 30%-70% relative humidity) for 30 minutes, then was cocured with the adhesive. Two different epoxy film adhesives were used in this evaluation: 3M Company's AF 163-2M (0.06 psf) and Henkel's Hysol EA 9696 (0.06 psf with mat carrier). Both adhesives were cured in a reusable vacuum bag apparatus under 15 inches Hg vacuum pressure.

Table 1: Sol-Gel Surface Preparation Details

Grit-blast /Sol-gel
* Acetone wipe with lint-free wipes * Scotch-Brite abrade using 3M VFN Roloc pads * Acetone wipe with Duralace 9404 wipes * Grit-blast using 50-micron alumina * Remove residual grit using 40-psi clean, dry filtered air * Apply AC-130 sol-gel for 3 minutes * Dry for 30 minutes at ambient temperature * If any areas are still wet, force dry panel with 10-psi clean, dry filtered air * Apply BR 6747-1 primer with a foam brush to a nominal thickenss of 0.2 mil * Dry for 30 minutes at ambient temperature * Apply adhesive * Cocure adhesive and primer for given cure cycle
Nylon-pad /Sol-gel
* Acetone wipe with Duralace 9404 wipes * Scotch-Brite abrade using 3M MED Roloc pads * Remove residual debris using 40-psi clean, dry filtered air * Apply AC-130 sol-gel for 3 minutes * Dry for 30 minutes at ambient temperature * If any areas are still wet, force dry panel with 10-psi clean, dry filtered air * Apply BR 6747-1 primer with a foam brush to a nominal thickenss of 0.2 mil * Dry for 30 minutes at ambient temperature * Apply adhesive * Cocure adhesive and primer for given cure cycle

Bond strength and durability were determined in this program using American Society for Testing and Materials (ASTM) standard test methods for tensile lap shear[10], floating roller peel[11], and wedge tests[12]. Wedge tests were conducted at two environmental conditions: (1) 120°F and 95-100% relative humidity (RH) and (2) 140°F and 95-100% RH. Tensile lap shear testing was conducted at ambient temperature (70°F) and 180°F. Floating roller peel testing was conducted at -65°F and ambient temperature (70°F). All specimens received a four-minute soak at temperature before testing.

2.1.3 Task A: Surface Preparation Evaluation

The purpose of Task A was to evaluate the effectiveness of the brush application process as part of three different surface preparations. PAA, grit-blast/sol-gel, and nylon pad/sol-gel, specimens were fabricated with AF 163-2M and EA 9696 adhesives and cured via the manufacturers' recommended cure cycle of 60 minutes at 250°F. The Task A test matrix is shown in Table 2. Five specimens per condition were tested. All specimens were fabricated with bare Al 2024-T3 stock.

Table 2: Task A Test Matrix

Surface Preparation		Wedge Test (ASTM D 3762)		Tensile Lap Shear (ASTM D 1002)		Floating Roller Peel (ASTM D 3167)	
		120°F & 98% RH	140°F & 98% RH	70°F	180°F	-65°F	70°F
AF 163-2M	PAA	5	5	5	5	5	5
	Grit-blast/sol-gel	5	5	5	5	5	5
	Nylon-pad/sol-gel	5	5	5	5	5	5
EA 9696	PAA	5	5	5	5	5	5
	Grit-blast/sol-gel	5	5	5	5	5	5
	Nylon-pad/sol-gel	5	5	5	5	5	5

2.1.4 Task B: Alternate Cure Evaluation

On-aircraft bonded repairs requiring elevated-temperature cure cycles routinely experience large temperature spreads due to uneven heating methods and heat sinks caused by large substructural components. Adhesive manufacturers suggest optimum cure cycles for their materials, but these are difficult to perform on aircraft. Therefore, an evaluation of lower-temperature cure cycles was conducted to determine the effect of those cures used in conjunction with the primer brush-application process. The alternate cure cycles of EA 9696 and AF 163-2M were determined via

differential scanning calorimetry (DSC) in previous work at AFRL/MLSA[13]. Table 3 contains a summary of the pertinent DSC test results from the previous work.

Table 3: DSC Results for AF 163-2M and EA 9696[13]

Cure Cycle	Property	AF 163-2M	EA 9696
1 hr @ 250°F	Time to max cure	33 minutes	25 minutes
	Tg [1]	234°F	239°F
	% Cure	97%	100%
6 hrs @ 200°F	Time to max cure	204 minutes	179 minutes
	Tg	243°F	232°F
	% Cure	89%	90%
8 hrs @ 180°F	Time to max cure	350 minutes	350 minutes
	Tg	207°F	207°F
	% Cure	76%	72%

[1] T_g = Glass Transition Temperature

<u>BR 6747-1 Alternate Cure Evaluation</u>

Since the BR 6747-1 primer was cocured with the adhesive in this program, alternate cure cycles also had to be defined for the primer. Solvent resistance testing was utilized to determine acceptable alternate cure cycles for BR 6747-1 primer. Al 2024-T3 0.063-inch sheet stock was machined into two-inch square specimens. The specimens were treated with PAA and primed with BR 6747-1 using the brush application method described in Figure 1. After a 30-minute dry at ambient laboratory conditions (70°F and 40% RH), the primer was exposed to various temperatures for given times in an air-circulating oven. The primed specimens then received fifty double-wipes with MEK-soaked, lint-free wipes. If the primer was unaffected by the MEK, the primer was deemed cured. If the MEK removed a visible portion of the primer, the primer was considered uncured.

<u>Determining the Effect of Alternate Cure Cycles on Brush-Primer Process</u>

Two low-temperature alternate cure cycles for cocuring the primer and adhesive were evaluated for use with the brush-on primer: 6 hours at 200°F and 8 hours at 180°F. Data from specimens cured according to these cure cycles were then compared to Task A data that utilized manufacturers' recommended cure of 60 minutes at 250°F for cocuring the adhesive and primer.

The Task B test matrix is shown in Table 4. The matrix was completed once using specimens prepared using nylon pad/sol-gel and once with specimens prepared via grit-blast/sol-gel.

Table 4: Task B Test Matrix

Cocure Cycle		Wedge Test (ASTM D 3762)		Tensile Lap Shear (ASTM D 1002)		Floating Roller Peel (ASTM D 3167)	
		120°F & 98% RH	140°F & 98% RH	70°F	180°F	-65°F	70°F
AF 163-2M	6 hrs @ 200°F	5	5	5	5	5	5
	8 hrs @ 180°F	5	5	5	5	5	5
EA 9696	6 hrs @ 200°F	5	5	5	5	5	5
	8 hrs @ 180°F	5	5	5	5	5	5

2.1.5 Results

Primer Thickness Evaluation Results

Primer thickness results for various color chips are shown in Table 5. In general, it appeared difficult to apply the primer in excess of Cytec's recommended upper limit for primer thickness of 0.4 mil (0.0004 inch). In fact, care was required to ensure the primer thickness met Cytec's minimum thickness requirement of 0.1 mil (0.0001 inch).

Table 5: BR 6747-1 Color Chip Primer Thickness Measurements

Specimen ID	Isoscope Average Measurements on PAA (mils) Range (minimum-maximum)	
	Side #1	Side #2
1	0.09 (0.04-0.11)	0.07 (0.05-0.10)
2	0.13 (0.08-0.17)	0.09 (0.04-0.15)
3	0.20 (0.08-0.24)	0.06 (0.03-0.09)
4	0.12 (0.07-0.16)	0.14 (0.07-0.19)

Task A: Surface Preparation Evaluation Results

Results for the tensile lap shear and floating roller peel testing for Task A are shown in Table 6. All lap shear specimens failed 100% cohesively within the adhesive layer regardless of surface preparation or adhesive type. Differences in lap shear strength are noticed between PAA, grit-blast/sol-gel, and nylon-pad/sol-gel specimens. These differences are assumed to be due to trapped moisture causing porosity in the bondline with the sol-gel surface preps. Fusing the primer using a heat gun prior to bonding could drive moisture off the adherend and eliminate some of the trapped moisture. Fusing is a process used to heat the dry primer powder coat to a

temperature sufficient the flow the primer over the surface. Previous work has shown that AF 163-2M tends to be more vacuum sensitive than EA 9696, thus the lower lap shear strengths achieved with AF 163-2M specimens[14]. All ambient temperature peel specimens failed cohesively, with little difference detected between surface preparations. Peel testing conducted at -65°F exhibited a range of failure modes.

Table 6: Task A Tensile Lap Shear and Floating Roller Peel Test Results

Surface Preparation		Lap Shear Strength (psi)				Peel Strength (pli)			
		70°F		180°F		-65°F		70°F	
AF 163-2M	PAA	4883	100% co	2235	100% co	52.1	100% co	66.8	100% co
AF 163-2M	Grit-blast/sol-gel	3401	100% co	1984	100% co	49.6	100% co	55.0	100% co
AF 163-2M	Nylon-pad/sol-gel (3M MED Scotch-Brite)	4002	100% co	2381	100% co	47.7	32% co	60.0	100% co
EA 9696	PAA	6070	100% co	4201	100% co	53.3	100% co	65.6	100% co
EA 9696	Grit-blast/sol-gel	3982	100% co	2815	100% co	44.6	100% co	56.7	100% co
EA 9696	Nylon-pad/sol-gel (3M MED Scotch-Brite)	4506	100% co	3562	100% co	55.3	52% co	55.1	100% co

co: cohesive failure

Results for wedge tests conducted at 120°F and 98% RH are shown in Table 7. All EA 9696 specimens failed 100% cohesively, regardless of surface preparation. Sol-gel specimens bonded with AF 163-2M exhibited a mix of cohesive failure and interfacial failure at the adhesive-primer interface, as verified through energy-dispersive (x-ray) spectrometry (EDS). Results of the wedge tests conducted at 140°F and 98% RH are shown in Table 8. As with testing performed at 120°F, all EA 9696 specimens failed cohesively while AF 163-2M sol-gel specimens exhibited a mix of cohesive failure and interfacial failure at the primer-adhesive interface, as verified via EDS. It should be noted that, although the sol-gel specimens exhibited interfacial failure at the primer-adhesive interface, the aluminum-primer interface was still intact and protected from the environment. The crack extension data for the sol-gel specimens were also very good, with crack growths of 0.20 inch or less after 28 days of exposure.

Table 7: Task A Wedge Test Results at 120°F & 98% RH

Adhesive	Surface Preparation	BLT** (in)	Initial (in)	Cumulative Crack Growth (in)							Failure Mode
				1 hr	8 hr	24 hr	7 days	14 days	21 days	28 days	
AF 163-2M	PAA	0.0060	1.28	0.00	0.01	0.02	0.03	0.04	0.04	0.04	98% co
AF 163-2M	Grit-blast/sol-gel	0.0060	1.29	0.03	0.03	0.03	0.05	0.05	0.05	0.08	67% co*
AF 163-2M	Nylon-pad/sol-gel (3M med)	0.0050	1.16	0.03	0.08	0.08	0.10	0.12	0.12	0.12	38% co*
EA 9696	PAA	0.0045	1.39	0.00	0.00	0.00	0.02	0.02	0.03	0.03	100% co
EA 9696	Grit-blast/sol-gel	0.0052	1.45	0.00	0.00	0.00	0.00	0.01	0.01	0.02	100% co
EA 9696	Nylon-pad/sol-gel (3M med)	0.0051	1.37	0.00	0.00	0.01	0.04	0.06	0.08	0.08	100% co

* remaining noncohesive failure occurred between primer and adhesive
** BLT: bondline thickness

Table 8: Task A Wedge Test Results at 140°F & 98% RH

Adhesive	Surface Preparation	BLT (in)	Initial (in)	Cumulative Crack Growth (in)							Failure Mode
				1 hr	8 hr	24 hr	7 days	14 days	21 days	28 days	
AF 163-2M	PAA	0.0053	1.29	0.00	0.03	0.04	0.04	0.05	0.06	0.09	100% co
AF 163-2M	Grit-blast/sol-gel	0.0058	1.31	0.00	0.01	0.02	0.04	0.11	0.11	0.14	88% co*
AF 163-2M	Nylon-pad/sol-gel (3M med)	0.0054	1.21	0.05	0.06	0.09	0.12	0.15	0.15	0.20	63% co*
EA 9696	PAA	0.0047	1.43	0.00	0.00	0.00	0.00	0.01	0.01	0.01	100% co
EA 9696	Grit-blast/sol-gel	0.0048	1.42	0.02	0.03	0.05	0.05	0.09	0.10	0.13	100% co
EA 9696	Nylon-pad/sol-gel (3M med)	0.0047	1.44	0.03	0.03	0.03	0.04	0.08	0.08	0.09	100% co

* remaining noncohesive failure occurred between primer and adhesive

<u>Task B: Alternate Cure Evaluation Results</u>

Alternate Cure Evaluation for BR 6747-1 Primer - Typical results of primer solvent-resistance testing for this effort are shown in Figure 3. Results of the primer alternate cure cycle testing are shown in Table 9. Testing demonstrated that BR 6747-1 is capable of curing to a solvent-resistant state at temperatures as low as 180°F. BR 6747-1 cured to a solvent-resistant state in the alternate cure cycles of 6 hours at 200°F and 8 hours at 180°F.

Failed Sample *Solvent Resistant Sample*

Figure 3: Typical Primer Alternate Cure Specimens

Table 9: Primer Alternate Cure Evaluation Results

Cure Temperature	Time of Exposure					
	1 hour	2 hours	3 hours	4 hours	6 hours	8 hours
250°F	cured					
225°F	cured	cured				
200°F	n-c	n-c	cured			
180°F	n-c	n-c	n-c	n-c	n-c	cured

n-c: not cured

Effect of Alternate Cure Cycles on Brush-Primer Process - Results of grit-blast/sol-gel lap shear and peel testing are shown in Table 10. All lap shear and peel specimens failed cohesively, regardless of adhesive type or cure cycle. No major differences in bond strength were detected due to alternate cure cycles, although EA 9696 specimens tended to exhibit higher lap shear strengths than AF 163-2M specimens at all test conditions.

Table 10: Effect of Alternate Cure Cycles on Lap Shear and Peel Strengths Using Grit-Blast/Sol-Gel with Brush Primer Application

Cure Cycles		Lap Shear Strength (psi)				Peel Strength (pli)			
		70°F		180°F		-65°F		70°F	
AF 163-2M	1 hr @ 250°F	3401	100% co	1984	100% co	49.6	100% co	55.0	100% co
	6 hrs @ 200°F	4322	100% co	1795	100% co	47.2	97% co	50.7	100% co
	8 hrs @ 180°F	3746	100% co	1783	100% co	46.3	100% co	45.0	100% co
EA 9696	1 hr @ 250°F	3982	100% co	2815	100% co	44.6	100% co	56.7	100% co
	6 hrs @ 200°F	4292	100% co	2558	100% co	48.4	99% co	57.4	100% co
	8 hrs @ 180°F	3776	100% co	2710	100% co	46.4	100% co	45.0	100% co

Results of the nylon pad/sol-gel lap shear and peel testing are shown in Table 11. All lap shear and peel specimens failed cohesively, regardless of adhesive type or cure cycle. No major differences in lap shear strength were detected due to alternate cure cycles, although EA 9696 specimens tended to exhibit higher lap shear strengths than AF 163-2M specimens at all testing conditions. All ambient-temperature peel specimens failed cohesively as well. However, a number of -65°F peel specimens failed in the primer layer, as verified through EDS. This failure mode was noticed in all AF 163-2M peel specimens but in only the control (cured for 60 minutes at 250°F) EA 9696 specimens. Primer layer failures in peel tests conducted at -65°F were also detected in prior testing for specimens primed with BR 6747-1 via spray gun and bonded with

AF 163-2M[15]. These failures occurred despite nominal primer thicknesses of 0.2 mil, well within the manufacturer's recommended limits.

Table 11: Effect of Alternate Cure Cycles on Lap Shear and Peel Strengths Using Nylon Pad/Sol-Gel with Brush Primer Application

Cure Cycles		Lap Shear Strength (psi)				Peel Strength (pli)			
		70°F		180°F		-65°F		70°F	
AF 163-2M	1 hr @ 250°F	4002	100% co	2381	100% co	47.7	32% co	60.0	100% co
	6 hrs @ 200°F	4414	100% co	3195	100% co	41.4	30% co	56.3	100% co
	8 hrs @ 180°F	5029	100% co	2788	100% co	26.6	24% co	52.3	100% co
EA 9696	1 hr @ 250°F	4506	100% co	3562	100% co	55.3	52% co	55.1	100% co
	6 hrs @ 200°F	5141	100% co	3255	100% co	49.8	100% co	55.3	100% co
	8 hrs @ 180°F	5103	100% co	3865	100% co	45.2	100% co	55.1	100% co

Results for grit-blast/sol-gel wedge tests conducted at 120°F and 98% RH are shown in Table 12. All EA 9696 specimens exhibited short crack growths after 28 days and failed cohesively. All AF 163-2M grit-blast/sol-gel specimens failed between the adhesive and primer (as verified through EDS), but yielded short crack growths similar to specimens failing cohesively. Results for the grit-blast/sol-gel wedge tests conducted at 140°F and 98% RH are shown in Table 13. These results were similar to the 120°F wedge test results since all EA 9696 grit-blast/sol-gel wedge test specimens failed cohesively and AF 163-2M grit-blast/sol-gel wedge test specimens exhibited failure between the primer and adhesive. The AF 163-2M grit-blast/sol-gel specimens also yielded short crack growths similar to wedge test specimens exhibiting cohesive failure modes. No differences were detected due to curing the adhesives using the lower-temperature alternate cure cycles when performing wedge tests at either 120°F or 140°F.

Table 12: Effect of Alternate Cure Cycles on 120°F Wedge Test Results Using Grit-Blast/Sol-Gel with Brush Primer Application

Cure Cycles		GLT (in)	Initial (in)	Cumulative Crack Growth (in)							Failure Mode
				1 hr	8 hr	24 hr	7 days	14 days	21 days	28 days	
AF 163-2M	1 hr @ 250°F	0.0060	1.29	0.03	0.03	0.03	0.05	0.05	0.05	0.08	67% co*
	6 hrs @ 200°F	0.0052	1.41	0.02	0.02	0.07	0.08	0.10	0.10	0.12	61% co*
	8 hrs @ 180°F	0.0067	1.46	0.02	0.03	0.04	0.06	0.07	0.07	0.08	95% co*
EA 9696	1 hr @ 250°F	0.0052	1.45	0.00	0.00	0.00	0.00	0.01	0.01	0.02	100% co
	6 hrs @ 200°F	0.0050	1.48	0.00	0.00	0.01	0.02	0.04	0.05	0.10	100% co
	8 hrs @ 180°F	0.0056	1.39	0.02	0.02	0.02	0.02	0.02	0.03	0.05	100% co

* remaining noncohesive failure occurred between primer and adhesive

Table 13: Effect of Alternate Cure Cycles on 140°F Wedge Test Results Using Grit-Blast/Sol-Gel with Brush Primer Application

Cure Cycles		GLT (in)	Initial (in)	Cumulative Crack Growth (in)							Failure Mode
				1 hr	8 hr	24 hr	7 days	14 days	21 days	28 days	
AF 163-2M	1 hr @ 250°F	0.0058	1.31	0.00	0.01	0.02	0.04	0.11	0.11	0.14	88% co*
	6 hrs @ 200°F	0.0056	1.38	0.03	0.08	0.10	0.12	0.16	0.16	0.18	85% co*
	8 hrs @ 180°F	0.0064	1.37	0.01	0.05	0.05	0.08	0.09	0.09	0.15	70% co*
EA 9696	1 hr @ 250°F	0.0048	1.42	0.02	0.03	0.05	0.05	0.09	0.10	0.13	100% co
	6 hrs @ 200°F	0.0045	1.49	0.01	0.02	0.02	0.06	0.10	0.11	0.15	100% co
	8 hrs @ 180°F	0.0046	1.53	0.03	0.03	0.03	0.05	0.05	0.05	0.08	100% co

* remaining noncohesive failure occurred between primer and adhesive

Results for the nylon pad/sol-gel wedge tests conducted at 120°F and 98% RH are shown in Table 14. All EA 9696 specimens exhibited short crack growths after 28 days and failed cohesively. All AF 163-2M grit-blast/sol-gel specimens exhibited failure between the adhesive and primer (as verified via EDS), but exhibited short crack growths similar to specimens failing cohesively. Results for the nylon pad/sol-gel wedge tests conducted at 140°F and 98% RH are shown in Table 15. These results were similar to the 120°F wedge test results since all EA 9696 grit-blast/sol-gel wedge test specimens failed cohesively and AF 163-2M grit-blast/sol-gel wedge test specimens exhibited failure between the primer and adhesive. The AF 163-2M grit-blast/sol-gel specimens also yielded short crack growths similar to wedge test specimens exhibiting cohesive failure modes.

Table 14: Effect of Alternate Cure Cycles on 120°F Wedge Test Results Using Nylon Pad/Sol-Gel With Brush Primer Application

Cure Cycles		GLT (in)	Initial (in)	Cumulative Crack Growth (in)							Failure Mode
				1 hr	8 hr	24 hr	7 days	14 days	21 days	28 days	
AF 163-2M	1 hr @ 250°F	0.0050	1.16	0.03	0.08	0.08	0.10	0.12	0.12	0.12	38% co*
	6 hrs @ 200°F	0.0052	1.32	0.02	0.06	0.09	0.12	0.13	0.13	0.14	47% co*
	8 hrs @ 180°F	0.0067	1.45	0.01	0.01	0.01	0.02	0.03	0.04	0.04	61% co*
EA 9696	1 hr @ 250°F	0.0051	1.37	0.00	0.00	0.01	0.04	0.06	0.08	0.08	100% co
	6 hrs @ 200°F	0.0050	1.36	0.01	0.02	0.02	0.06	0.07	0.09	0.10	91% co
	8 hrs @ 180°F	0.0051	1.43	0.00	0.00	0.00	0.04	0.04	0.06	0.07	100% co

* remaining noncohesive failure occurred between primer and adhesive

Table 15: Effect of Alternate Cure Cycles on 140°F Wedge Test Results Using Nylon Pad/Sol-Gel With Brush Primer Application

Cure Cycles		GLT (in)	Initial (in)	Cumulative Crack Growth (in)							Failure Mode
				1 hr	8 hr	24 hr	7 days	14 days	21 days	28 days	
AF 163-2M	1 hr @ 250°F	0.0054	1.21	0.05	0.06	0.09	0.12	0.15	0.15	0.20	63% co*
	6 hrs @ 200°F	0.0052	1.34	0.08	0.11	0.13	0.16	0.22	0.22	0.24	89% co*
	8 hrs @ 180°F	0.0051	1.45	0.03	0.04	0.04	0.05	0.09	0.09	0.12	39% co*
EA 9696	1 hr @ 250°F	0.0047	1.44	0.03	0.03	0.03	0.04	0.08	0.08	0.09	100% co
	6 hrs @ 200°F	0.0051	1.44	0.00	0.05	0.05	0.09	0.14	0.14	0.14	98% co
	8 hrs @ 180°F	0.0053	1.42	0.02	0.05	0.07	0.07	0.11	0.11	0.16	98% co

* remaining noncohesive failure occurred between primer and adhesive

2.1.6 Conclusions

The brush application technique evaluated in this study appears to be an effective way to apply BR 6747-1 adhesive bond primer. The technique requires no specialized equipment or difficult processing steps. A variety of different-sized foam brushes purchased at several home improvement/hardware stores were successfully used to apply BR 6747-1 primer to PAA, grit-blast/sol-gel, and nylon pad/sol-gel surfaces with coating thicknesses verified in the desired range of 0.1-0.2 mil (0.0001-0.0002 inch). In fact, unlike spray application techniques, applying thick primer coatings outside the manufacturer's specified range (>0.4 mil) proved to be more difficult with the brush application techniques.

Task A testing showed that using the brush application process to apply BR 6747-1 to PAA, grit-blat/sol-gel, and nylon pad/sol-gel surfaces did not reduce lap shear strength, peel strength, or performance in the wedge test. Specimens bonded with EA 9696 tended to exhibit higher lap shear strengths than those bonded with AF 163-2M, possibly due to greater adverse effects of vacuum curing on the AF 163-2M adhesive. AF 163-2M wedge test specimens exhibited interfacial failure between the adhesive and primer. Since failure did not occur at the aluminum-primer interface or within the primer layer, it is not suspected to be a surface preparation or brush application deficiency, especially since this same phenomenon has been witnessed in past work using a spray application method to apply the primer[15].

Task B testing identified alternate cure cycles at 200°F and 180°F for AF 163-2M, EA 9696, and BR 6747-1. No differences in lap shear strength, peel strength, or bond environmental durability (wedge test performance) were detected due to brush application of the

primer, alternate cure cycles, or a combination of using the brush application method with alternate low-temperature cure cycles. It is interesting to note although curing AF 163-2M and EA 9696 at 180°F reduced the percent cure to ~75% and the T_g of the systems to 207°F (Table 3), there seemed to be no detriment to bond strength or durability when lower-temperature adhesive and primer cure cycles were used with either grit-blast/sol-gel or nylon-pad/sol-gel surface preparations.

The successful combination of a convenient brush application technique for BR 6747-1 and effective low-temperature cure cycles for AF 163-2M, EA 9696, and BR 6747-1 provides aircraft maintainers with a useful tool for performing on-component bonded repairs. The brush application process is safer to perform than spray techniques since chromates are not dispersed into the air. Brush-application is also advantageous in the field environment because specialized equipment such as spray guns, clean air supply, and personal protection equipment (respirators) are not required. Since the brush-application technique is safer and easier to perform in the field, it reduces the amount of time required to perform a bonded repair while at the same time providing a strong, durable adhesive bond.

2.2 EFFECT OF VACUUM HOLDS

Performing a bonded repair from start to finish without stopping can be difficult in the field or depot due to time constraints. Typically, performing the surface preparation can be accomplished in a single work shift, but curing the adhesive can extend the repair time into a second shift. Due to elevated costs associated with operating a second shift, depot personnel requested an evaluation to determine if the sol-gel prepared surface, adhesive, and repair can be sealed in a vacuum bag and held for up to 24 hours prior to curing the adhesive. This process would allow greater flexibility in the bond shops.

2.2.1 Test Plan

The use of vacuum holds was evaluated using Henkel’s Hysol EA 9696 (0.06 psf) and Hysol EA 9628 (0.06 psf) adhesives with cocured Cytec BR 6747-1 primer. Several surface preparations

were evaluated, including phosphoric acid anodize, grit-blast/sol-gel (GBSG), and nylon pad/sol-gel (NPSG). Tensile lap shear testing[10] was conducted at ambient temperature (70°F) using Al 7075-T6 adherends.

Surface Preparations

Phosphoric Acid Anodize - Adherends composed of Al 7075-T6 were phosphoric acid anodized (PAA)[8]. Adherends were primed using a high velocity, low-pressure (HVLP) spray gun using either Cytec BR 127 or BR 6747-1 then dried at ambient laboratory conditions (70°F & 40% RH) for 30 minutes. BR 127 was always precured for 60 minutes at 250°F prior to bonding. Adherends primed with BR 6747-1 were cocured with the adhesive, fused with a heat gun then cocured with the adhesive, or precured for 60 minutes at 250°F.

Grit-Blast/Sol-Gel (GBSG) - GBSG specimens were degreased using acetone-soaked, lint-free wipes until all visible traces of contamination were removed. Adherends were abraded with VFN Scotch-Brite Roloc pads using a 20,000 RPM pneumatic grinder to achieve a baseline surface. Adherends were grit-blasted using 50-micron Al_2O_3 grit then blown with 35 psi N_2 to remove loose grit from the bond surface. The bond surfaces were wetted for 3 minutes with AC-130 sol-gel[16] using an acid brush. Adherends were orientated vertically and dried for 30 minutes at ambient laboratory conditions. Once dried, adherends were primed with BR 6747-1 sprayed using an HVLP gun. Primed adherends were cocured with the adhesive, fused with a heat gun then cocured with the adhesive, or precured for 60 minutes at 250°F.

Nylon-Pad/Sol-Gel (NPSG) - NPSG specimens were degreased using acetone-soaked, lint-free wipes until all visible traces of contamination were removed. Adherends were abraded with VFN Scotch-Brite Roloc pads using a 20,000 RPM pneumatic grinder to achieve a baseline surface and solvent degreased using acetone-soaked, lint-free wipes. A final abrasion was performed using MED Scotch-Brite Roloc pads, then adherends were blown with 35 psi N_2 to remove loose debris from the bond surface. The bond surfaces were wetted for 3 minutes with AC-130 sol-gel using an acid brush. Adherends were orientated vertically and dried for 30 minutes at ambient laboratory conditions. Once dried, adherends were primed with BR 6747-1 using the foam brush process described in Section 2.1. Primed adherends were

cocured with the adhesive, fused with a heat gun then cocured with the adhesive, or precured for 60 minutes at 250°F.

Vacuum Holds

In order to determine the effect of a vacuum hold, panels were sealed in a vacuum bag and placed under 15 or 20 inches Hg vacuum pressure prior to cure. In addition, a control panel was fabricated for each condition and cured immediately without a vacuum hold. In order to evaluate a worst-case scenario, vacuum holds were of 24-hour duration.

Adhesive Cure Cycles

Hysol EA 9628 and Hysol EA 9696 were both cured for 1 hour at 250°F and 15 in Hg vacuum pressure. Panels were cured in an air-circulating oven. A lap shear panel was fabricated using PAA and GBSG for each adhesive and cured under 35 psi positive pressure.

Tensile Lap Shear Test Procedures

All lap shear panels were machined into 1-inch wide specimens using a gang cutting mill. Specimens were not conditioned in any fashion. All specimens were tested per ASTM D 1002 at ambient laboratory conditions using a cross-head speed of 0.05 in/min.

2.2.2 Test Results

Results for EA 9696 testing are shown in Table 16. All failure modes were 100% cohesive. In general, the PAA-treated specimens failed at higher strengths than the sol-gel treated specimens. However, there was very little difference noticed between similarly treated specimens due to the use of a vacuum hold as compared to specimens bonded immediately upon completion of the surface preparation. This was true for PAA, GBSG, and NPSG specimens. Therefore, it is not believed the vacuum hold affects bond strength when cocuring BR 6747-1 with EA 9696 film adhesive.

Table 16: Effect of Vacuum Holds on Tensile Lap Shear Strength of EA 9696 Adhesive

Surface Preparation	Primer	Primer Cure	Vacuum Hold	Cure Pressure	GLT (in)	RT Lap Shear Strength (psi) [std dev]
PAA	BR 127	Precured	none	35 psi	0.0041	6302 [238]
PAA	BR 127	Precured	none	15 in Hg	0.0033	6144 [79]
PAA	BR 127	Precured	24 hrs @ 15 in Hg	15 in Hg	0.0033	5521 [67]
PAA	BR 127	Precured	24 hrs @ 25 in Hg	15 in Hg	0.0032	6311 [109]
PAA	BR 6747-1	Precured	none	35 psi	0.0041	6304 [160]
PAA	BR 6747-1	Precured	none	15 in Hg	0.0042	5635 [66]
PAA	BR 6747-1	Precured	24 hrs @ 15 in Hg	15 in Hg	0.0029	6023 [208]
PAA	BR 6747-1	Precured	24 hrs @ 25 in Hg	15 in Hg	0.0052	5610 [80]
PAA	BR 6747-1	Cocure	none	15 in Hg	0.0036	6429 [140]
PAA	BR 6747-1	Cocure	24 hrs @ 15 in Hg	15 in Hg	0.0044	6277 [272]
PAA	BR 6747-1	Cocure	24 hrs @ 25 in Hg	15 in Hg	0.0045	6310 [161]
PAA	BR 6747-1	Fusing / cocure	none	15 in Hg	0.0035	6282 [104]
PAA	BR 6747-1	Fusing / cocure	24 hrs @ 15 in Hg	15 in Hg	0.0037	6005 [174]
PAA	BR 6747-1	Fusing / cocure	24 hrs @ 25 in Hg	15 in Hg	0.0037	6242 [170]
GBSG	BR 6747-1	Precured	none	15 in Hg	0.0045	5163 [96]
GBSG	BR 6747-1	Precured	24 hrs @ 15 in Hg	15 in Hg	0.0032	5578 [215]
GBSG	BR 6747-1	Precured	24 hrs @ 25 in Hg	15 in Hg	0.0036	5641 [46]
GBSG	BR 6747-1	Cocure	none	15 in Hg	0.0042	4429 [118]
GBSG	BR 6747-1	Cocure	24 hrs @ 15 in Hg	15 in Hg	0.0032	5026 [124]
GBSG	BR 6747-1	Cocure	24 hrs @ 25 in Hg	15 in Hg	0.0049	5134 [106]
GBSG	BR 6747-1	Fusing / cocure	none	15 in Hg	0.0031	4632 [182]
GBSG	BR 6747-1	Fusing / cocure	24 hrs @ 15 in Hg	15 in Hg	0.0031	5073 [171]
GBSG	BR 6747-1	Fusing / cocure	24 hrs @ 25 in Hg	15 in Hg	0.0039	5011 [101]
NPSG	BR 6747-1	Cocure	none	15 in Hg	0.0038	5314 [166]
NPSG	BR 6747-1	Cocure	24 hrs @ 15 in Hg	15 in Hg	0.0049	5484 [105]
NPSG	BR 6747-1	Cocure	24 hrs @ 25 in Hg	15 in Hg	0.0032	5695 [121]
NPSG	BR 6747-1	Fusing / cocure	none	15 in Hg	0.0034	5372 [144]
NPSG	BR 6747-1	Fusing / cocure	24 hrs @ 15 in Hg	15 in Hg	0.0051	5137 [107]
NPSG	BR 6747-1	Fusing / cocure	24 hrs @ 25 in Hg	15 in Hg	0.0042	5349 [262]

PAA: Phosphoric Acid Anodize
GBSG: Grit-Blast/Sol-Gel
NPSG: Nylon Pad/Sol-Gel

Results for EA 9628 testing are shown in Table 17. All failure modes were 100% cohesive. Similar to specimens bonded with EA 9696, little difference was noticed due to the use of vacuum holds when cocuring BR 6747-1 with EA 9628.

Table 17: Effect of Vacuum Holds on Tensile Lap Shear Strength of EA 9628 Adhesive

Surface Preparation	Primer	Primer Cure	Vacuum Hold	Cure Pressure	GLT (in)	RT Lap Shear Strength (psi)	[std dev]
PAA	BR 127	Precured	none	35 psi	0.0043	5948	[46]
PAA	BR 127	Precured	none	15 in Hg	0.0030	6428	[133]
PAA	BR 127	Precured	24 hrs @ 15 in Hg	15 in Hg	0.0046	5650	[168]
PAA	BR 127	Precured	24 hrs @ 25 in Hg	15 in Hg	0.0049	6003	[119]
PAA	BR 6747-1	Precured	none	35 psi	0.0051	5938	[57]
PAA	BR 6747-1	Precured	none	15 in Hg	0.0049	5614	[96]
PAA	BR 6747-1	Precured	24 hrs @ 15 in Hg	15 in Hg	0.0029	6189	[55]
PAA	BR 6747-1	Precured	24 hrs @ 25 in Hg	15 in Hg	0.0040	6335	[259]
PAA	BR 6747-1	Cocure	none	15 in Hg	0.0050	6114	[126]
PAA	BR 6747-1	Cocure	24 hrs @ 15 in Hg	15 in Hg	0.0029	6831	[189]
PAA	BR 6747-1	Cocure	24 hrs @ 25 in Hg	15 in Hg	0.0031	6623	[80]
PAA	BR 6747-1	Fusing / cocure	none	15 in Hg	0.0027	6409	[157]
PAA	BR 6747-1	Fusing / cocure	24 hrs @ 15 in Hg	15 in Hg	0.0038	6132	[210]
PAA	BR 6747-1	Fusing / cocure	24 hrs @ 25 in Hg	15 in Hg	0.0028	6692	[242]
GBSG	BR 6747-1	Precured	none	15 in Hg	0.0037	5682	[156]
GBSG	BR 6747-1	Precured	24 hrs @ 15 in Hg	15 in Hg	0.0040	5925	[101]
GBSG	BR 6747-1	Precured	24 hrs @ 25 in Hg	15 in Hg	0.0044	5668	[54]
GBSG	BR 6747-1	Cocure	none	15 in Hg	0.0042	4872	[109]
GBSG	BR 6747-1	Cocure	24 hrs @ 15 in Hg	15 in Hg	0.0043	5195	[112]
GBSG	BR 6747-1	Cocure	24 hrs @ 25 in Hg	15 in Hg	0.0029	5527	[282]
GBSG	BR 6747-1	Fusing / cocure	none	15 in Hg	0.0035	4932	[241]
GBSG	BR 6747-1	Fusing / cocure	24 hrs @ 15 in Hg	15 in Hg	0.0049	4934	[61]
GBSG	BR 6747-1	Fusing / cocure	24 hrs @ 25 in Hg	15 in Hg	0.0037	5396	[105]
NPSG	BR 6747-1	Cocure	none	15 in Hg	0.0041	5447	[107]
NPSG	BR 6747-1	Cocure	24 hrs @ 15 in Hg	15 in Hg	0.0031	5467	[148]
NPSG	BR 6747-1	Cocure	24 hrs @ 25 in Hg	15 in Hg	0.0049	5986	[101]
NPSG	BR 6747-1	Fusing / cocure	none	15 in Hg	0.0031	5456	[272]
NPSG	BR 6747-1	Fusing / cocure	24 hrs @ 15 in Hg	15 in Hg	0.0045	5584	[96]
NPSG	BR 6747-1	Fusing / cocure	24 hrs @ 25 in Hg	15 in Hg	0.0053	5588	[92]

PAA: Phosphoric Acid Anodize
GBSG: Grit-Blast/Sol-Gel
NPSG: Nylon Pad/Sol-Gel

2.2.3 Conclusions

No appreciable difference was noticed in tensile lap shear strength due to the insertion of a vacuum hold in the bonding process when cocuring BR 6747-1 with EA 9628 or EA 9696 film adhesives. This was determined to be true for PAA, GBSG, and NPSG prepared Al 7075-T6. In addition, little difference in bond strength was noticed when utilizing different vacuum levels both during the hold and during the cure cycle. The use of a 24-hour vacuum hold does not appear to degrade lap shear strength for any of the evaluated surface pretreatments or primer/adhesive cure combinations.

2.3 EVALUATION OF HOT-WET LAP SHEAR PROPERTIES

Previous work proved that bonds prepared with the grit-blast and nylon pad sol-gel surface preparations without use of a bond primer were susceptible to degradation in a lap shear test at 180°F after exposure to 140°F and 98% relative humidity (RH)[17]. Specifically, the specimens failed 100% at the interface between the adhesive and aluminum substrate. This phenomenon was unexpected since the same surface preparations were evaluated with the same adhesives in the wedge test and aged at 120°F and 98% RH for 28 days. The wedge tests specimens failed 95-100% cohesively (within the adhesive), implying the use of bond primer was not critical for passing the wedge test. However, the drastic reduction in strength combined with interfacial failure modes in a hot/wet lap shear test reaffirm that the wedge test alone is not sufficient for determining the susceptibility of adhesive bonds to moisture. Additionally, these results present a possible issue with the susceptibility of sol-gel treated specimens to hot/wet environments. One possibility for this was the absence of Cytec BR 6747-1 bond primer from the process. A project was initiated to further evaluate the effect of hot/wet environmental exposures and testing on sol-gel treated aluminum adherends.

2.3.1 Test Program

Two evaluations were conducted in this program: (1) an evaluation to determine the baseline lap shear strength of specimens treated using various surface preparation procedures and (2) an evaluation to investigate the effects of sol-gel drying steps and primer cure cycles on lap shear

strengths. Sol-gel drying and primer cure cycles were evaluated to determine if trapped moisture was reducing lap shear strength. All specimens were fabricated with bare Al 2024-T3 adherends.

Baseline Lap Shear Strength Evaluation

The general test matrix for the baseline lap shear strength evaluation is shown in Table 18. This matrix was completed once using EA 9696 adhesive and once using AF 163-2M adhesive.

Table 18: Baseline Lap Shear Strength Evaluation Test Matrix

Surface Prep	# of Specimens per Test Condition		
	RT	180°F-Dry	180°F-Wet*
PAA	5	5	5
GB/SG	10	10	10
NP/SG	10	10	10

* wet specimens aged for 60 days at 140°F & 98% RH prior to testing

Surface Preparations - Three surface preparations were used prior to adhesive bonding: (1) phosphoric acid anodize, (2) grit-blast/sol-gel, and (3) nylon-pad/sol-gel. Full descriptions of the three surface preparations are shown below.

Specimens prepared via phosphoric acid anodize were initially solvent degreased using acetone-soaked, lint-free wipes. Adherends were wet-abraded using 3M Company 7447 Scotch-Brite pads for deoxidization. Adherends were dried in an air-circulating oven for 30 minutes at 160°F. Adherends were then anodized per ASTM D 3933 and rinsed in tap water for 5 minutes at RT (70°F). Adherends were dried in an air-circulating oven for 30 minutes at 160°F. Cytec BR 6747-1 adhesive bond primer was applied to a nominal thickness of 0.2 mil (0.0002 in) and dried at RT for 30 minutes. The primer was cured for 60 minutes at 250°F in an air-circulating oven.

Specimens prepared via grit-blast/sol-gel were initially solvent degreased using acetone-soaked, lint-free wipes. Adherends were abraded to a baseline surface using a VFN Scotch-Brite Roloc pad using a 20,000 RPM Dotco grinder driven by a clean air supply. The abraded surfaces were cleaned with acetone-soaked, lint-free wipes until no visible trace of contamination was present. The adherends were grit-blasted using 50μm Al_2O_3 to a matte finish. Adherends were blown with 35 psi clean air to remove any loose grit from the bond surface. AC-130 sol-gel was

applied to the grit-blasted surfaces using an acid brush so the surfaces were wetted for 3 minutes. The adherends were orientated vertically to drain and dry for 30 minutes. BR 6747-1 primer was applied via spray gun to a nominal thickness of 0.2 mil (0.0002 inch). Primer was dried for 30 minutes at RT (70°F). The primer was cocured with the adhesive in a portable autoclave.

Specimens prepared via nylon pad/sol-gel were initially solvent degreased using acetone-soaked lint-free wipes. Adherends were abraded with a MED Scotch-Brite Roloc pad using a 20,000 RPM Dotco grinder driven by a clean air supply. The abraded surfaces were blown with 35 psi clean air to remove any loose debris from the bond surface. AC-130 sol-gel was applied to the abraded surfaces using an acid brush so the surfaces were wetted for 3 minutes. The adherends were orientated vertically to drain and dry for 30 minutes. BR 6747-1 primer was applied via spray gun to a nominal thickness of 0.2 mil (0.0002 inch). Primer was dried for 30 minutes at RT (70°F). The primer was cocured with the adhesive in a portable autoclave.

Lap Shear Specimen Fabrication - Lap shear panels were bonded together using one of two epoxy film adhesives: 3M Company AF 163-2M (0.06 psf) or Hysol EA 9696 (0.06 psf) from Henkel. Adhesive and primer were cocured in a portable autoclave for 1 hour at 250°F under 35 psi positive pressure. Lap shear panels were machined into 1-inch wide specimens using a gang cutting mill. Bondline thicknesses were measured using an optical microscope.

Lap Shear Specimen Conditioning and Testing - When required, specimens were aged at 140°F and 98% RH for 60 days prior to testing. Specimens were soaked for 10 minutes prior to testing at 180°F (dry) and soaked for 4 minutes prior to testing at 180°F (wet). All testing was performed according to ASTM D 1002.

Effect of Sol-Gel Drying and Primer Cure Methods

The general test matrix for the sol-gel drying and primer cure evaluation is shown in Table 19. The matrix was conducted once using EA 9696 adhesive and once using AF 163-2M adhesive.

Table 19: Sol-Gel Drying and Primer Cure Evaluation Test Matrix

Surface Prep	Sol-Gel Drying	Primer Cure	# of Specimens/Condition	
			180°F-Dry	180°F-Wet*
GB/SG	30 minutes @ 220°F	Precure	5	5
	30 minutes @ 220°F	Cocure	5	5
	30 minutes @ RT	Cocure	5	5
NP/SG	30 minutes 220°F	Precure	5	5
	30 minutes @ 220°F	Cocure	5	5
	30 minutes @ RT	Cocure	5	5

* wet specimens aged for 60 days at 140°F & 98% RH prior to testing

Surface Preparations - Variations of the grit-blast/sol-gel and nylon pad/sol-gel surface preparations were evaluated to determine the effect of sol-gel drying cycles and primer cure cycles on lap shear strength.

Specimens prepared via grit-blast/sol-gel were initially solvent degreased using acetone-soaked, lint-free wipes. Adherends were abraded to a baseline surface with a VFN Scotch-Brite Roloc pad using a 20,000 RPM Dotco grinder driven by a clean air supply. The abraded surfaces were cleaned with acetone-soaked, lint-free wipes until no visible trace of contamination was present. The adherends were grit-blasted using 50μm Al_2O_3 to a matte finish. Adherends were blown with 35 psi clean air to remove any loose grit from the bond surface. AC-130 sol-gel was applied to the grit-blasted surfaces using an acid brush so the surfaces were wetted for 3 minutes. The adherends were orientated vertically to drain and dry for 30 minutes. Some specimens were also dried at 220°F for 30 minutes in an air-circulating. BR 6747-1 primer was applied via spray gun to a nominal thickness of 0.2 mil (0.0002 inch). Primer was dried for 30 minutes at RT (70°F). The primer was either precured in an air circulating oven for 60 minutes at 250°F or cocured with the adhesive in a portable autoclave.

Specimens prepared via nylon pad/sol-gel were initially solvent degreased using acetone-soaked, lint-free wipes. Adherends were abraded with a MED Scotch-Brite Roloc pad using a 20,000 RPM Dotco grinder driven by a clean air supply. The abraded surfaces were blown with 35 psi clean air to remove any loose debris from the bond surface. AC-130 sol-gel was applied to the abraded surfaces using an acid brush so the surfaces were wetted for 3 minutes. The adherends were orientated vertically to drain and dry for 30 minutes. Some specimens were also dried at 220°F for 30 minutes in an air-circulating oven. BR 6747-1 primer was applied via

spray gun to a nominal thickness of 0.2 mil (0.0002 inch). Primer was dried for 30 minutes at RT (70°F). The primer was either precured in an air circulating oven for 60 minutes at 250°F or cocured with the adhesive in a portable autoclave.

Lap Shear Specimen Fabrication **-** Lap shear panels were bonded using one of two epoxy film adhesives: 3M Company AF 163-2M (0.06 psf) or Hysol EA 9696 (0.06 psf) from Henkel. Adhesive and primer were cocured in a portable autoclave for 1 hour at 250°F under 35 psi positive pressure. Lap shear panels were machined into 1-inch wide specimens using a gang cutting mill. Bondline thicknesses were measured using an optical microscope.

Lap Shear Specimen Conditioning and Testing - When required, specimens were aged at 140°F and 98% RH for 60 days prior to testing. Specimens were soaked for 10 minutes prior to testing at 180°F (dry) and soaked for 4 minutes prior to testing at 180°F (wet). All testing was performed according to ASTM D 1002.

2.3.2 Test Results

Baseline Lap Shear Test Results

Results of the baseline lap shear testing using EA 9696 are shown in Table 20. Failure modes are given in percentage of cohesive failure. The remaining amount of failure occurred between the aluminum and primer. Two values are given for each set of GB/SG and NP/SG treated specimens. These values are the averages of two separate lap shear panels (10 specimens total). Only one lap shear panel was fabricated using PAA. In general, the PAA specimens exhibit the highest strengths followed by the NP/SG specimen strengths and lastly the GB/SG specimen strengths. Even though the NP/SG specimens typically have higher strengths than GB/SG specimens, the GB/SG specimens typically exhibit higher percentages of cohesive failure. One possible cause could have been trapped moisture on the blasted surface creating bondline porosity. This small porosity was observed under microscopic analysis and is shown in Figure 4.

Table 20: Baseline Lap Shear Strengths of EA 9696

Surface Prep	Lap Shear Strength (psi) [% cohesive failure]		
	RT	180°F-Dry	180°F-Wet
PAA	6331 [100% co]	5014 [92% co]	3866 [92% co]
GB/SG	5255 [100% co]	4088 [100% co]	2075 [75% co]
	5186 [100% co]	3592 [100% co]	2019 [89% co]
NP/SG	5725 [100% co]	4871 [60% co]	3167 [36% co]
	5855 [100% co]	4251 [74% co]	2488 [32% co]

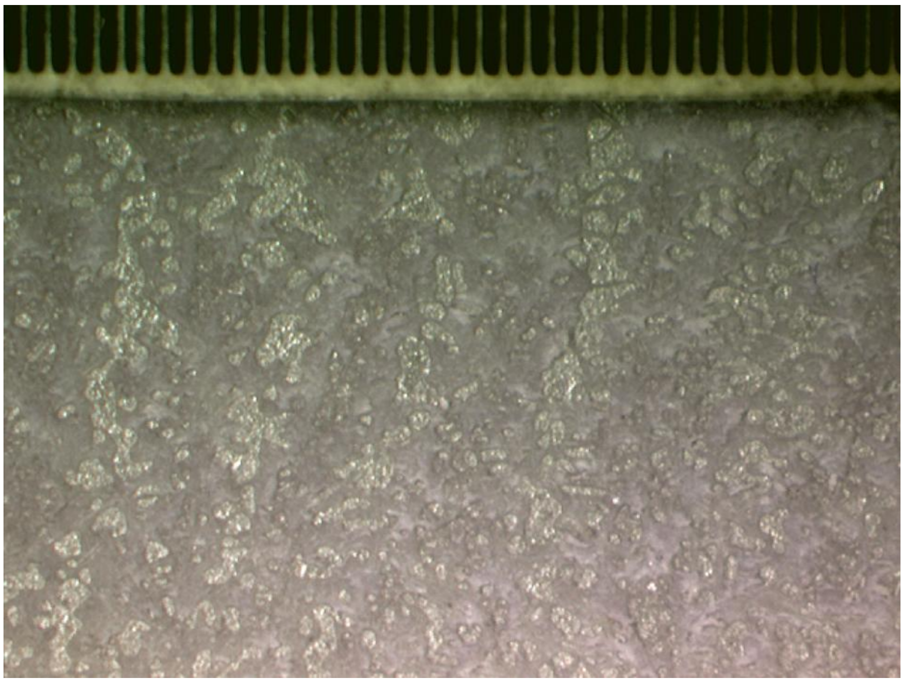


Figure 4: Porosity Observed in a Grit-Blast/Sol-Gel Lap Shear Specimen Bonded with EA 9696 Film Adhesive Under an Optical Microscope

Results of the baseline lap shear testing using AF 163-2M are shown in Table 21. Failure modes are given in percentage of cohesive failure. The remaining amount of failure occurred between the aluminum and primer. Two values are given for each set of GB/SG and NP/SG treated specimens. These values are the averages of two separate lap shear panels (10 specimens total). Only one lap shear panel was fabricated using PAA. As observed with EA 9696, the PAA specimens exhibit the highest strengths followed by the NP/SG specimen strengths and lastly the GB/SG specimen strengths. Even though the NP/SG specimens typically have higher strengths

than GB/SG specimens, the GB/SG specimens typically exhibit higher percentages of cohesive failure. As before, one possible cause could have been trapped moisture creating porosity. This small porosity was observed under microscopic analysis.

Table 21: Baseline Lap Shear Strengths of AF 163-2M

Surface Prep	Lap Shear Strength (psi) [% cohesive failure]		
	RT	180°F-Dry	180°F-Wet
PAA	5483 [100% co]	4036 [100% co]	3053 [96% co]
GB/SG	4811 [100% co]	3203 [75% co]	1833 [81% co]
	4374 [100% co]	2849 [100% co]	1465 [100% co]
NP/SG	5077 [100% co]	3950 [30% co]	2659 [42% co]
	5276 [100% co]	3749 [100% co]	1832 [16% co]

Sol-Gel Drying and Primer Cure Evaluation Test Results

Results of the EA 9696 lap shear testing for the sol-gel drying and primer cure evaluation are shown in Table 22. Drying the sol-gel at 220°F or precuring the primer appears to increase the lap shear strengths of GB/SG treated specimens. No difference is noticed in the NP/SG treated specimen strengths. Results of the AF 163-2M lap shear testing for the sol-gel drying and primer cure evaluation are shown in Table 23. Drying the sol-gel at 220°F or precuring the primer increased the lap shear strengths of GB/SG treated specimens. No difference was noticed in the NP/SG treated specimen strengths.

Table 22: Effect of Sol-Gel Drying and Primer Cures on EA 9696 Lap Shear Strength

Surface Prep	Sol-Gel Drying	Primer Cure	Lap Shear Strength (psi) [% cohesive failure]	
			180°F-Dry	180°F-Wet*
GB/SG	30 minutes @ 220°F	Precure	4276 [100% co]	3071 [82% co]*
	30 minutes @ 220°F	Cocure	4356 [100% co]	2721 [88% co]*
	30 minutes @ RT	Cocure	3507 [95% co]*	1885 [96% co]*
NP/SG	30 minutes 220°F	Precure	4232 [80% co]*	3178 [62% co]**
	30 minutes @ 220°F	Cocure	4262 [58% co]**	3018 [52% co]**
	30 minutes @ RT	Cocure	4582 [81% co]**	2880 [67% co]**

*   Noncohesive failure occurred between primer and adhesive

** Noncohesive failure occurred between aluminum and primer

Table 23: Effect of Sol-Gel Drying and Primer Cures on AF 163-2M Lap Shear Strength

Surface Prep	Sol-Gel Drying	Primer Cure	Lap Shear Strength (psi) [% cohesive failure]	
			180°F-Dry	180°F-Wet*
GB/SG	30 minutes @ 220°F	Precure	3598 [96% co]*	2529 [88% co]*
	30 minutes @ 220°F	Cocure	3593 [100% co]	2027 [68% co]*
	30 minutes @ RT	Cocure	3090 [100% co]	1536 [92% co]*
NP/SG	30 minutes 220°F	Precure	3902 [97% co]*	2199 [34% co]**
	30 minutes @ 220°F	Cocure	3908 [100% co]	2147 [22% co]**
	30 minutes @ RT	Cocure	3576 [100% co]	2241 [60% co]**

* Noncohesive failure occurred between primer and adhesive

** Noncohesive failure occurred between aluminum and primer

2.3.3 Conclusions

Lap shear testing at 180°F after 60 days exposure to 140°F & 98% RH proved that sol-gel treated specimens are still somewhat susceptible to moisture degradation. Interfacial failures are more prevalent in the NP/SG treated surfaces than the GB/SG treated surfaces. However, GB/SG treated surfaces are more likely to trap moisture on the surface, causing weaker adhesive (cohesive) strengths. The best combination of interfacial durability and adhesive strength appeared to be achievable when employing a sol-gel dry step at 220°F or precuring the primer prior to adhesive cure with the GB/SG surface preparation. This process yielded 180°F-wet lap shear strengths higher than strengths achieved when using a RT dry step and cocuring the adhesive and primer.

2.4 EFFECT OF AMBIENT TEMPERATURE AND HUMIDITY

A majority of the sol-gel surface preparation optimization to date was conducted in a controlled laboratory environment at 70°F & 40% RH. However, these processes need to be usable all over the world in environments that are not well controlled. This project was conducted in order to evaluate the effects of ambient temperature and humidity conditions on wedge test and lap shear test results for a number of different sol-gel surface preparation variations. In order to properly control the ambient temperature and humidity, all surface preparations were performed in the controlled environmental booth at the Coatings Technology Integration Office (AFRL/MLSSO) at Wright-Patterson Air Force Base, Ohio. This facility allowed for the temperature and humidity in a room to be varied so that specimens could be fabricated at alternate conditions. Specifically, four different conditions were evaluated:

1. 77°F & 50% RH,
2. 90°F & 10% RH,
3. 90°F & 85% RH and
4. 50°F & 85% RH.

Data obtained in this effort helped to better define processing parameters and help ensure that sol-gel treated repairs will perform well in a field environment.

2.4.1 Test Plan

The general test matrix is shown in Table 24. All specimens were fabricated from Al 2024-T3. This matrix was repeated using the five different surface preparations described below. A total of ten specimens per condition were fabricated. Five lap shear and five wedge test specimens were treated and bonded on one day and the remaining five specimens for each test were treated and bonded the following day for a total of ten. This process was used throughout the effort, so all specimens for a given temperature and humidity condition were fabricated over a two day period.

Table 24: Ambient Temperature and Humidity Effects Test Matrix

Sol-Gel Drying	Lap Shear RT	Wedge Test 120°F & 98% RH
Blow dry with N_2 (Blow dry)	10	10
Until visibly dry (Appears dry)	10	10
30 minutes @ RT	10	10

Specimen Fabrication

Five surface preparation/adhesive combinations were evaluated: (1) NP/SG without primer using Hysol EA 9320NA paste adhesive, (2) NP/SG with BR 6747-1 cocured with EA 9696 film adhesive, (3) NP/SG with BR 6747-1 fused and cocured with EA 9696, (4) GB/SG with BR 6747-1 cocured with EA 9696, and (5) GB/SG with BR 6747-1 cocured with EA 9696. These processes are described in detail in the following sections. Once the adherends were treated, panels were assembled and sealed in a vacuum bag in the environmental chamber. The bagged parts were then transported under vacuum to AFRL/MLSA and immediately cured in an air-circulating oven. Once cured, panels were machined into 1-inch wide specimens and tested.

NP/SG without Primer Using EA 9320NA Paste Adhesive - Specimens were degreased using acetone-soaked, lint-free wipes until clean and abraded using MED 3M Scotch-Brite Roloc pads. The bond surfaces were blown with filtered N_2 to remove any abrasion debris from the surface. AC-130 sol-gel was applied with an acid brush so the surfaces were kept wet for three minutes. The sol-gel treated surfaces were dried using one of the methods described in Table 24. Specimens were bonded with EA 9320NA paste adhesive. Glass beads were mixed with the adhesive (0.5% by weight of the resin) for bondline control. Specimens were bonded at 180°F and 15 inches Hg vacuum pressure in an air-circulating oven.

NP/SG with BR 6747-1 Cocured with EA 9696 Film Adhesive - Specimens were degreased using acetone-soaked, lint-free wipes until clean and abraded using MED 3M Scotch-Brite Roloc pads. The bond surfaces were blown with filtered N_2 to remove any abrasion debris from the surface. AC-130 sol-gel was applied with an acid brush so the surfaces were kept wet for 3 minutes. The sol-gel treated surfaces were dried using one of the methods described in Table 24. BR 6747-1 bond primer was applied using a foam brush, and the specimens were dried for 30 minutes at ambient conditions. The primer and EA 9696 film adhesive were cocured at 250°F and 15 inches Hg vacuum pressure in an air-circulating oven.

***NP/SG with BR 6747-1 Fused then Cocured with EA 9696 Film Adhesive* -** Specimens were degreased using acetone-soaked, lint-free wipes until clean and abraded using MED 3M Scotch-Brite Roloc pads. The bond surfaces were blown with filtered N_2 to remove any abrasion debris from the surface. AC-130 sol-gel was applied with an acid brush so the surfaces were kept wet for 3 minutes. The sol-gel treated surfaces were dried using one of the methods described in Table 24. BR 6747-1 bond primer was applied using a foam brush, and the specimens were dried for 30 minutes at ambient conditions. After the 30-minute ambient dry, a heat gun was used to fuse the primer. It was estimated that the temperature of the air from the heat gun was between 250°F-300°F and required approximately five minutes to complete the process. EA 9696 film adhesive was applied once the panels cooled to ambient temperature and the primer and adhesive were cocured at 250°F and 15 inches Hg vacuum pressure in an air-circulating oven.

GB/SG with BR 6747-1 Cocured with EA 9696 Film Adhesive **-** Specimens were degreased using acetone-soaked, lint-free wipes until clean and abraded using VFN 3M Scotch-Brite Roloc pads. The bond surfaces were blown with filtered N_2 to remove any abrasion debris from the surface. The bond surfaces were grit-blasted using 50 μm Al_2O_3. Residual grit was removed using 35 psi filtered N_2. AC-130 sol-gel was applied with an acid brush so the surfaces were kept wet for 3 minutes. The sol-gel treated surfaces were dried using one of the methods described in Table 24. BR 6747-1 bond primer was applied using a foam brush and the specimens were dried for 30 minutes at ambient conditions. The primer and EA 9696 film adhesive were cocured at 250°F and 15 inches Hg vacuum pressure in an air-circulating oven.

GB/SG with BR 6747-1 Fused then Cocured with EA 9696 Film Adhesive **-** Specimens were degreased using acetone-soaked, lint-free wipes until clean and abraded using VFN 3M Scotch-Brite Roloc pads. The bond surfaces were blown with filtered N_2 to remove any abrasion debris from the surface. The bond surfaces were grit-blasted using 50 μm Al_2O_3. Residual grit was removed using 35 psi filtered N_2. AC-130 sol-gel was applied with an acid brush so the surfaces were kept wet for 3 minutes. The sol-gel treated surfaces were dried using one of the methods described in Table 24. BR 6747-1 bond primer was applied using a foam brush and the specimens were dried for 30 minutes at ambient conditions. After the 30-minute ambient dry, a heat gun was used to fuse the primer. It was estimated that the temperature of the air from the heat gun was between 250°F-300°F and required approximately 5 minutes to complete to process. EA 9696 film adhesive was applied once the panels cooled to ambient temperature and the primer and adhesive were cocured at 250°F under 15 inches Hg vacuum pressure in an air-circulating oven.

<u>Specimen Conditioning and Testing</u>

Lap shear specimens received no conditioning prior to testing at ambient laboratory conditions (70°F and 40% RH). Wedge test specimens were exposed to 120°F and 98% RH for 28 days. Crack growth was monitored throughout the test. After 28 days, the wedge test specimens were removed from the test chamber and opened to determine failure modes. Failure modes were reported as the percentage of the test area exhibiting cohesive failure within the adhesive layer.

2.4.2 Results

Results are presented in this section and grouped together depending on surface treatment performed during processing.

NP/SG without Primer Using EA 9320NA Paste Adhesive

Wedge test results for NP/SG treated specimens bonded with EA 9320NA are shown in Table 25. Overall, results are very inconsistent with previously reported results using this adhesive[18]. There also did not appear to be many trends witnessed in the wedge test results due to sol-gel drying method or ambient conditions. One possible reason for the poor results could be the limited working life of the EA 9320NA paste adhesive. The paste adhesive only had a reduced working time at elevated temperature and started to set. It was difficult to get the parts sealed in a vacuum bag and transported to the AFRL/MLSA laboratory prior to the adhesive achieving a gelled state. Oftentimes, vacuum was lost during transportation and the adhesive set prior to restoring vacuum pressure at MLSA. Poor test results, likely due to the adhesive setting prior to application of vacuum, was most noticeable when fabricating specimens at the 90°F conditions.

Table 25: Effect of Ambient Conditions and Sol-Gel Dry Method on Bond Durability for Nylon-Pad/Sol-Gel without Bond Primer using EA 9320NA Adhesive

Ambient Conditions	Sol-Gel Dry Method	Initial (in)	Cumulative Crack Growth (in)							Total (in)	Failure Mode
			1 hr	8 hr	24 hr	7 day	14 day	21 day	28 day		
50°F / 85% RH	Blown dry	1.41	0.13	0.16	0.22	0.51	0.61	0.71	0.76	2.18	8% co
		1.67	0.07	0.09	0.09	0.12	0.20	0.24	0.30	1.97	10% co
	30 minutes	1.50	0.06	0.18	0.27	0.60	0.75	0.84	0.90	2.39	10% co
		1.47	0.09	0.16	0.25	0.42	0.49	0.51	0.54	2.01	-0-% co
	46 minutes	1.52	0.09	0.19	0.34	0.67	0.81	0.91	0.95	2.47	10% co
		1.55	0.07	0.10	0.19	0.48	0.55	0.62	0.65	2.20	-0-% co
77°F / 50% RH	Blown dry	1.60	0.06	0.10	0.14	0.37	0.48	0.55	0.59	2.19	31% co
		1.79	0.03	0.06	0.08	0.25	0.30	0.35	0.39	2.18	6% co
	12 minutes	1.95	0.10	0.17	0.23	0.49	0.60	0.63	0.63	2.58	79% co
		1.61	0.05	0.10	0.15	0.30	0.45	0.48	0.50	2.11	-0-% co
	30 minutes	2.27	0.09	0.28	0.55	0.69	0.76	0.80	0.82	3.09	98% co
		1.56	0.09	0.18	0.31	0.56	0.75	0.77	0.79	2.35	2% co
90°F / 10% RH	Blown dry	1.74	0.02	0.02	0.03	0.15	0.19	0.25	0.28	2.02	6% co
		1.88	0.01	0.01	0.01	0.01	0.08	0.14	0.16	2.04	85% co
	8 minutes	1.95	0.01	0.01	0.03	0.12	0.14	0.19	0.22	2.17	18% co
		1.75	0.01	0.01	0.03	0.06	0.08	0.19	0.20	1.95	46% co
	30 minutes	1.94	0.00	0.00	0.01	0.05	0.07	0.11	0.13	2.07	40% co
		1.72	0.00	0.02	0.03	0.13	0.22	0.32	0.33	2.05	6% co
90°F / 85% RH	Blown dry	1.81	0.15	0.33	0.48	0.67	0.93	0.96	0.97	2.78	55% co
		1.75	0.17	0.34	0.41	0.59	0.59	0.73	0.75	2.50	76% co
	22 minutes	1.72	0.07	0.20	0.40	0.61	0.73	0.73	0.76	2.48	42% co
		1.97	0.11	0.35	0.42	0.56	0.63	0.69	0.70	2.67	74% co
	30 minutes	2.17	0.21	0.37	0.40	0.66	0.77	0.80	0.82	2.99	67% co
		1.71	0.09	0.16	0.18	0.30	0.33	0.37	0.41	2.12	56% co

Lap shear results for NP/SG treated specimens bonded with EA 9320NA are shown in Table 26. A graphical interpretation of these data are shown in Figure 5. Two sets of lap shear specimens experienced a vacuum leak during transport to MLSA resulting in a loss of consolidation pressure. Due to the reduced working life of the adhesive, the EA 9320NA set up prior to reapplication of vacuum pressure and poor bonds resulted. When comparing the results obtained at different temperature/humidity conditions, the lowest strengths were obtained when processing the specimens at 90°F and 85% RH. This is also the condition where most moisture was present in the air and where it proved most difficult to adequately dry the specimens. None of the specimens tested in this effort met the strength published in the Hysol EA 9320NA technical data sheet of 4600 psi[19]. This could be due to a number of factors, the most likely being: (1) the strengths published in the Hysol technical data sheet were generated using positive pressure on PAA prepared adherends while the strengths presented in this report were

generated using vacuum pressure on a NP/SG surface preparation, and/or (2) gelling of the adhesive prior to the final application of bond pressure during final cure.

Table 26: Effect of Ambient Conditions and Sol-Gel Dry Method on Tensile Lap Shear Strength for Nylon-Pad/Sol-Gel without Bond Primer using EA 9320NA Adhesive

Surface Preparation	Environmental Conditions	Sol-Gel Dry Method	Lap Shear Strength (psi) (Failure Mode)	
			Trial #1	Trial #2
Nylon pad/sol-gel without bond primer, EA 9320NA with Glass Beads	50°F / 85% RH	Blown dry	3505 (64% co)	4343 (90% co)
	50°F / 85% RH	46 minutes	3783 (72% co)	4322 (90% co)
	50°F / 85% RH	30 minutes	3688 (74% co)	4070 (80% co)
	77°F / 50% RH	Blown dry	4129 (85% co)	3590 (92% co)
	77°F / 50% RH	12 minutes	4409 (80% co)	No consolidation
	77°F / 50% RH	30 minutes	4444 (90% co)	4072 (87% co)
	90°F / 10% RH	Blown dry	3674 (73% co)	4353 (90% co)
	90°F / 10% RH	8 minutes	3763 (88% co)	4060 (82% co)
	90°F / 10% RH	30 minutes	3808 (83% co)	3857 (80% co)
	90°F / 85% RH	Blown dry	3511 (90% co)	3090 (90% co)
	90°F / 85% RH	22 minutes	2415 (88% co)	3883 (95% co)
	90°F / 85% RH	30 minutes	3266 (89% co)	No consolidation

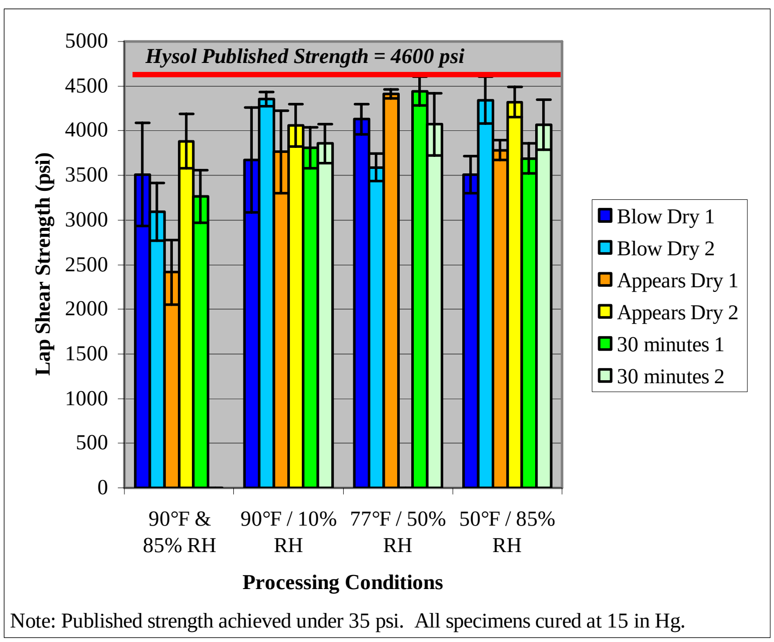


Figure 5: Lap Shear Strength Versus Processing Conditions for NP/SG without Primer Bonded with EA 9320NA

NP/SG with BR 6747-1 Cocured with EA 9696 Film Adhesive

Wedge test results for NP/SG treated specimens with BR 6747-1 cocured with EA 9696 are shown in Table 27. Wedge test results were more consistent than those achieved with the paste adhesive. This could be due to the fact that the NP/SG treatment performs better in the wedge test with bond primer, but it is likely due to the fact that there are no difficulties similar to the paste adhesive specimens associated with transporting the film adhesive specimens. Almost all failure modes were 90% cohesive or higher except when processing specimens at the conditions with 85% RH.

Table 27: Effect of Ambient Conditions and Sol-Gel Dry Method on Bond Durability for Nylon-Pad/Sol-Gel with BR 6747-1 Cocured with EA 9696 Adhesive

Ambient Conditions	Sol-Gel Dry Method	Initial (in)	Cumulative Crack Growth (in)							Total (in)	Failure Mode
			1 hr	8 hr	24 hr	7 day	14 day	21 day	28 day		
50°F / 85% RH	Blown dry	1.30	0.10	0.11	0.15	0.23	0.25	0.25	0.27	1.57	82% co
		1.27	0.10	0.12	0.13	0.16	0.19	0.19	0.20	1.47	95% co
	30 minutes	1.30	0.09	0.10	0.11	0.21	0.23	0.25	0.28	1.58	72% co
		1.28	0.09	0.13	0.13	0.20	0.20	0.25	0.25	1.52	77% co
	46 minutes	1.28	0.06	0.06	0.07	0.13	0.15	0.19	0.19	1.47	93% co
		1.26	0.03	0.08	0.10	0.16	0.17	0.17	0.18	1.44	94% co
77°F / 50% RH	Blown dry	1.29	0.04	0.07	0.11	0.14	0.15	0.18	0.19	1.48	94% co
		1.26	0.01	0.06	0.06	0.13	0.15	0.17	0.18	1.44	92% co
	12 minutes	1.18	0.03	0.06	0.07	0.12	0.13	0.16	0.17	1.35	95% co
		1.31	0.02	0.04	0.04	0.05	0.08	0.11	0.11	1.42	94% co
	30 minutes	1.23	0.03	0.06	0.06	0.12	0.14	0.14	0.16	1.39	93% co
		1.22	0.03	0.06	0.06	0.11	0.12	0.14	0.17	1.39	95% co
90°F / 10% RH	Blown dry	1.31	0.04	0.06	0.06	0.10	0.11	0.12	0.12	1.44	95% co
		1.31	0.01	0.03	0.05	0.08	0.11	0.11	0.11	1.42	95% co
	8 minutes	1.22	0.06	0.10	0.12	0.15	0.15	0.16	0.16	1.38	94% co
		1.27	0.03	0.05	0.08	0.09	0.09	0.09	0.11	1.38	93% co
	30 minutes	1.31	0.01	0.03	0.03	0.09	0.10	0.10	0.11	1.42	95% co
		1.29	0.01	0.02	0.03	0.03	0.06	0.06	0.06	1.35	95% co
90°F / 85% RH	Blown dry	1.23	0.07	0.07	0.08	0.12	0.17	0.17	0.17	1.41	92% co
		1.22	0.07	0.07	0.10	0.13	0.15	0.16	0.18	1.40	95% co
	22 minutes	1.30	0.06	0.07	0.09	0.13	0.19	0.20	0.21	1.51	91% co
		1.15	0.07	0.12	0.13	0.19	0.21	0.25	0.25	1.40	93% co
	30 minutes	1.28	0.05	0.05	0.08	0.14	0.16	0.18	0.19	1.47	88% co
		1.25	0.02	0.05	0.08	0.15	0.15	0.17	0.18	1.43	95% co

Lap shear results for NP/SG treated specimens with BR 6747-1 cocured with EA 9696 are shown in Table 28. A graphical interpretation of these data is shown in Figure 6. When comparing the results obtained at different temperature/humidity conditions, the lowest strengths were obtained when processing the specimens at 90°F and 85% RH. This is also the condition where most

moisture was present in the air and where it proved most difficult to adequately dry the specimens. Conversely, the highest strengths were obtained when processing the specimens at 90°F and 10% RH. This was the condition most beneficial for drying the sol-gel. None of the specimens tested in this effort met the UDRI published strengths of 5709 psi[20] when cured under 20 in Hg. The higher value was generated using PAA surface preparation, whereas the numbers presented in this report were generated using a NP/SG surface preparation.

Table 28: Effect of Ambient Conditions and Sol-Gel Dry Method on Tensile Lap Shear Strength for Nylon-Pad/Sol-Gel with BR 6747-1 Cocured with EA 9696 Adhesive

Surface Preparation	Environmental Conditions	Sol-Gel Dry Method	Lap Shear Strength (psi) (Failure Mode)	
			Trial #1	Trial #2
Nylon-Pad/Sol-Gel with BR 6747-1 Cocured Primer, EA 9696 Adhesive	50°F / 85% RH	Blown dry	4326 (75% co)	4800 (80% co)
	50°F / 85% RH	46 minutes	3861 (42% co)	4207 (60% co)
	50°F / 85% RH	30 minutes	3486 (30% co)	4745 (75% co)
	77°F / 50% RH	Blown dry	4334 (90% co)	4926 (85% co)
	77°F / 50% RH	12 minutes	4257 (93% co)	4416 (78% co)
	77°F / 50% RH	30 minutes	4484 (92% co)	4434 (82% co)
	90°F / 10% RH.	Blown dry	4984 (85% co)	4443 (80% co)
	90°F / 10% RH.	8 minutes	4612 (88% co)	5428 (92% co)
	90°F / 10% RH.	30 minutes	5037 (95% co)	5614 (95% co)
	90°F / 85% RH	Blown dry	2274 (10% co)	4108 (78% co)
	90°F / 85% RH	22 minutes	2111 (10% co)	3373 (32% co)
	90°F / 85% RH	30 minutes	3535 (40% co)	3392 (66% co)

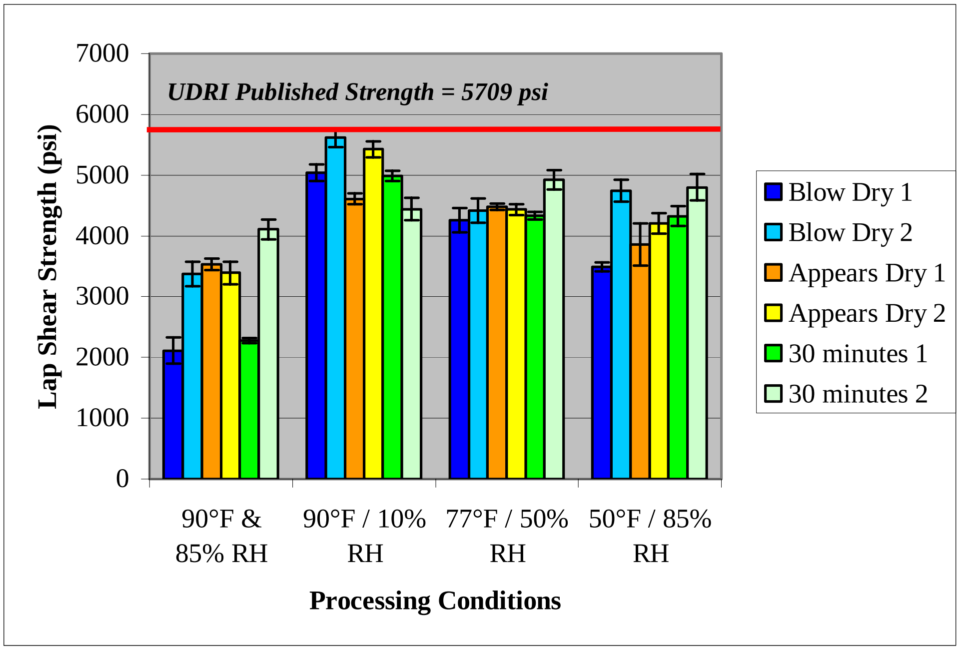


Figure 6: Lap Shear Strength Versus Processing Conditions for NP/SG with BR 6747-1 Cocured with EA 9696

NP/SG with BR 6747-1 Fused then Cocured with EA 9696 Film Adhesive

Wedge test results for NP/SG treated specimens with fused BR 6747-1 cocured with EA 9696 are shown in Table 29. Wedge test results were more consistent than those achieved with the paste adhesive. This could be due to the fact that the NP/SG treatment performs better in the wedge test with bond primer, but is more likely due to the fact that there are no difficulties, similar to the paste adhesive specimens, associated with transporting the film adhesive specimens. All the failure modes were 90% cohesive or higher except for a single set of specimens processing at 50°F and 85% RH.

Table 29: Effect of Ambient Conditions and Sol-Gel Dry Method on Bond Durability for Nylon-Pad/Sol-Gel with Fused BR 6747-1 Cocured with EA 9696 Adhesive

Ambient Conditions	Sol-Gel Dry Method	Initial (in)	Cumulative Crack Growth (in)							Total (in)	Failure Mode
			1 hr	8 hr	24 hr	7 day	14 day	21 day	28 day		
50°F / 85% RH	Blown dry	1.28	0.05	0.05	0.08	0.15	0.19	0.20	0.21	1.49	87% co
		1.30	0.04	0.07	0.08	0.12	0.14	0.16	0.16	1.46	95% co
	30 minutes	1.33	0.05	0.07	0.07	0.18	0.19	0.21	0.22	1.55	93% co
		1.42	0.02	0.07	0.07	0.12	0.19	0.19	0.20	1.62	95% co
	46 minutes	1.31	0.09	0.09	0.09	0.15	0.19	0.20	0.20	1.52	95% co
		1.34	0.04	0.04	0.08	0.11	0.12	0.15	0.15	1.49	95% co
77°F / 50% RH	Blown dry	1.24	0.07	0.09	0.10	0.13	0.14	0.15	0.16	1.40	95% co
		1.18	0.09	0.09	0.09	0.13	0.15	0.15	0.17	1.35	95% co
	12 minutes	1.21	0.05	0.08	0.11	0.13	0.16	0.17	0.18	1.39	95% co
		1.21	0.04	0.08	0.08	0.14	0.14	0.17	0.18	1.39	95% co
	30 minutes	1.18	0.05	0.10	0.12	0.15	0.16	0.16	0.20	1.38	94% co
		1.25	0.03	0.06	0.07	0.12	0.16	0.16	0.19	1.44	95% co
90°F / 10% RH	Blown dry	1.24	0.04	0.09	0.09	0.11	0.15	0.16	0.16	1.40	94% co
		1.31	0.04	0.07	0.09	0.10	0.11	0.12	0.14	1.45	94% co
	8 minutes	1.25	0.03	0.05	0.05	0.10	0.11	0.12	0.14	1.39	94% co
		1.29	0.04	0.06	0.06	0.06	0.07	0.11	0.11	1.40	95% co
	30 minutes	1.27	0.02	0.03	0.06	0.09	0.09	0.12	0.13	1.40	95% co
		1.28	0.00	0.02	0.07	0.08	0.09	0.12	0.12	1.40	93% co
90°F / 85% RH	Blown dry	1.30	0.08	0.08	0.11	0.15	0.19	0.20	0.21	1.51	91% co
		1.22	0.06	0.10	0.10	0.13	0.15	0.17	0.18	1.40	94% co
	22 minutes	1.29	0.05	0.05	0.06	0.13	0.17	0.18	0.18	1.47	94% co
		1.30	0.07	0.08	0.10	0.15	0.19	0.19	0.19	1.49	95% co
	30 minutes	1.33	0.03	0.06	0.06	0.11	0.15	0.15	0.16	1.49	93% co
		1.26	0.07	0.11	0.11	0.16	0.19	0.21	0.23	1.47	94% co

Lap shear results for NP/SG treated specimens with fused BR 6747-1 cocured with EA 9696 are shown in Table 30. A graphical interpretation of these data is shown in Figure 7. When comparing the results obtained at different temperature/humidity conditions, the lowest strengths were obtained when processing the specimens at 90°F and 85% RH. This is also the condition where most moisture was present in the air and where it proved most difficult to adequately dry the specimens. Conversely, the highest strengths were obtained when processing the specimens at 90°F and 10% RH. This was the condition most beneficial for drying the sol-gel. None of the specimens tested in this effort met the UDRI published strength of 5709 psi. Additionally, the shear strength values obtained using the primer fuse step appear to be higher than those obtained when cocuring without the fuse step. This is noticeable at the more humid application conditions such as 90°F and 85% RH as well as 50°F and 85% RH. The data also seem to be more

consistent when comparing drying processes. This is likely due to the use of a primer fuse step with a heat gun prior to adhesive cure. This step helps remove trapped moisture on the surface.

Table 30: Effect of Ambient Conditions and Sol-Gel Dry Method on Tensile Lap Shear Strength for Nylon-Pad/Sol-Gel with Fused BR 6747-1 Cocured with EA 9696 Adhesive

Surface Preparation	Environmental Conditions	Sol-Gel Dry Method	Lap Shear Strength (psi) (Failure Mode)	
			Trial #1	Trial #2
Nylon-Pad/Sol-Gel with BR 6747-1 Fused Primer, EA 9696 Adhesive	50°F / 85% RH	Blown dry	3627 (25% co)	4432 (77% co)
	50°F / 85% RH	46 minutes	2977 (36% co)	4091 (58% co)
	50°F / 85% RH	30 minutes	3500 (50% co)	3081 (60% co)
	77°F / 50% RH	Blown dry	3220 (20% co)	4389 (80% co)
	77°F / 50% RH	12 minutes	3296 (20% co)	4246 (70% co)
	77°F / 50% RH	30 minutes	3403 (60% co)	4498 (80% co)
	90°F / 10% RH.	Blown dry	5236 (90% co)	5068 (85% co)
	90°F / 10% RH.	8 minutes	4923 (85% co)	Fixture problem
	90°F / 10% RH.	30 minutes	5374 (94% co)	5001 (86% co)
	90°F / 85% RH	Blown dry	3841 (50% co)	4089 (64% co)
	90°F / 85% RH	22 minutes	3672 (40% co)	3910 (60% co)
	90°F / 85% RH	30 minutes	3742 (36% co)	3883 (44% co)

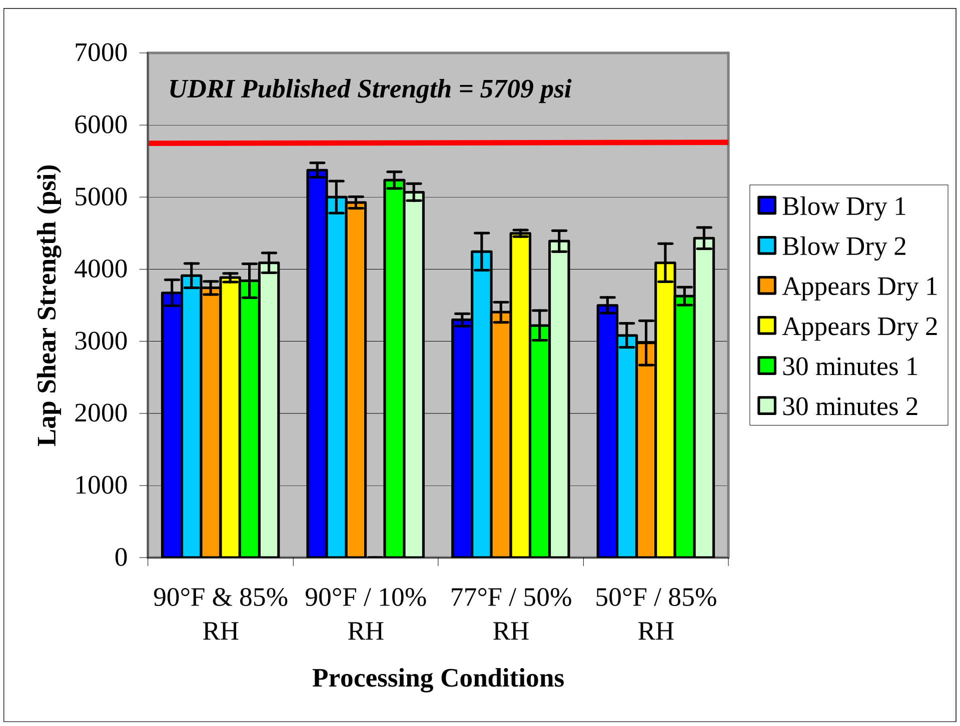


Figure 7: Lap Shear Strength Versus Processing Conditions for NP/SG with Fused BR 6747-1 Cocured with EA 9696 Adhesive

GB/SG with BR 6747-1 Cocured with EA 9696 Film Adhesive

Wedge test results for GB/SG treated specimens with BR 6747-1 cocured with EA 9696 are shown in Table 31. Wedge test results were very consistent, routinely obtaining >90% cohesive failure. Only a few sets of specimens exhibited less than 90% cohesive failure. The remaining noncohesive failure occurred at the primer-adhesive interface.

Table 31: Effect of Ambient Conditions and Sol-Gel Dry Method on Bond Durability for Grit-Blast/Sol-Gel with BR 6747-1 Cocured with EA 9696 Adhesive

Ambient Conditions	Sol-Gel Dry Method	Initial (in)	Cumulative Crack Growth (in)							Total (in)	Failure Mode
			1 hr	8 hr	24 hr	7 day	14 day	21 day	28 day		
50°F / 85% RH	Blown dry	1.35	0.06	0.08	0.09	0.19	0.22	0.23	0.23	1.58	80% co
		1.29	0.08	0.10	0.11	0.17	0.17	0.21	0.21	1.51	95% co
	30 minutes	1.27	0.05	0.05	0.07	0.14	0.15	0.18	0.19	1.47	79% co
		1.32	0.01	0.05	0.06	0.10	0.13	0.14	0.14	1.46	95% co
	46 minutes	1.36	0.03	0.05	0.05	0.13	0.14	0.14	0.14	1.50	95% co
		1.33	0.00	0.03	0.10	0.14	0.15	0.17	0.17	1.53	95% co
77°F / 50% RH	Blown dry	1.24	0.07	0.07	0.07	0.14	0.14	0.14	0.15	1.39	96% co
		1.30	0.02	0.02	0.05	0.10	0.10	0.12	0.14	1.44	95% co
	12 minutes	1.25	0.06	0.09	0.11	0.17	0.19	0.20	0.20	1.45	95% co
		1.29	0.00	0.04	0.04	0.04	0.14	0.14	0.15	1.44	95% co
	30 minutes	1.21	0.05	0.09	0.09	0.15	0.16	0.17	0.17	1.38	95% co
		1.27	0.00	0.03	0.05	0.10	0.10	0.17	0.17	1.44	94% co
90°F / 10% RH	Blown dry	1.30	0.07	0.09	0.09	0.15	0.17	0.17	0.18	1.48	95% co
		1.33	0.03	0.07	0.07	0.07	0.08	0.12	0.12	1.46	91% co
	8 minutes	1.26	0.07	0.09	0.10	0.14	0.15	0.15	0.15	1.41	95% co
		1.33	0.01	0.03	0.05	0.07	0.10	0.10	0.10	1.43	89% co
	30 minutes	1.31	0.04	0.07	0.07	0.14	0.14	0.15	0.16	1.47	95% co
		1.37	0.04	0.07	0.09	0.09	0.14	0.15	0.15	1.52	95% co
90°F / 85% RH	Blown dry	1.22	0.07	0.08	0.08	0.15	0.18	0.18	0.19	1.41	89% co
		1.32	0.05	0.07	0.07	0.15	0.15	0.16	0.17	1.49	93% co
	22 minutes	1.31	0.04	0.07	0.07	0.12	0.18	0.18	0.18	1.50	91% co
		1.31	0.05	0.06	0.09	0.15	0.15	0.18	0.21	1.52	95% co
	30 minutes	1.28	0.08	0.11	0.11	0.15	0.18	0.19	0.21	1.49	91% co
		1.27	0.05	0.06	0.08	0.12	0.12	0.15	0.16	1.43	95% co

Lap shear results for GB/SG treated specimens with BR 6747-1 cocured with EA 9696 are shown in Table 32. A graphical interpretation of these data is shown in Figure 8. When comparing the results obtained at different temperature/humidity conditions, the lowest strengths were obtained when processing the specimens at 90°F and 85% RH or 50°F and 85% RH. These conditions were the environments with the largest amounts of moisture present in the air and where it proved most difficult to adequately dry the specimens. Conversely, the highest strengths were obtained when processing the specimens at 90°F and 10% RH as well as 77°F and 50% RH. These conditions

proved to be drier and more beneficial for drying the sol-gel. None of the specimens tested in this effort met the UDRI published strength of 5709 psi.

Table 32: Effect of Ambient Conditions and Sol-Gel Dry Method on Tensile Lap Shear Strength for Grit-Blast/Sol-Gel with BR 6747-1 Cocured with EA 9696 Adhesive

Surface Preparation	Environmental Conditions	Sol-Gel Dry Method	Lap Shear Strength (psi) (Failure Mode)	
			Trial #1	Trial #2
Grit-Blast/Sol-Gel with BR 6747-1 Cocured Primer, EA 9696 Adhesive	50°F / 85% RH	Blown dry	2516 (30% co)	2960 (40% co)
	50°F / 85% RH	46 minutes	3229 (40% co)	2693 (30% co)
	50°F / 85% RH	30 minutes	2408 (20% co)	4232 (80% co)
	77°F / 50% RH	Blown dry	4393 (70% co)	4304 (90% co)
	77°F / 50% RH	12 minutes	4596 (90% co)	4168 (88% co)
	77°F / 50% RH	30 minutes	4531 (85% co)	3968 (95% co)
	90°F / 10% RH.	Blown dry	4554 (90% co)	4184 (90% co)
	90°F / 10% RH.	8 minutes	3047 (95% co)	4146 (88% co)
	90°F / 10% RH.	30 minutes	4642 (90% co)	4546 (93% co)
	90°F / 85% RH	Blown dry	2092 (20% co)	2306 (20% co)
	90°F / 85% RH	22 minutes	2542 (20% co)	3493 (80% co)
	90°F / 85% RH	30 minutes	2642 (20% co)	2570 (-0-% co)

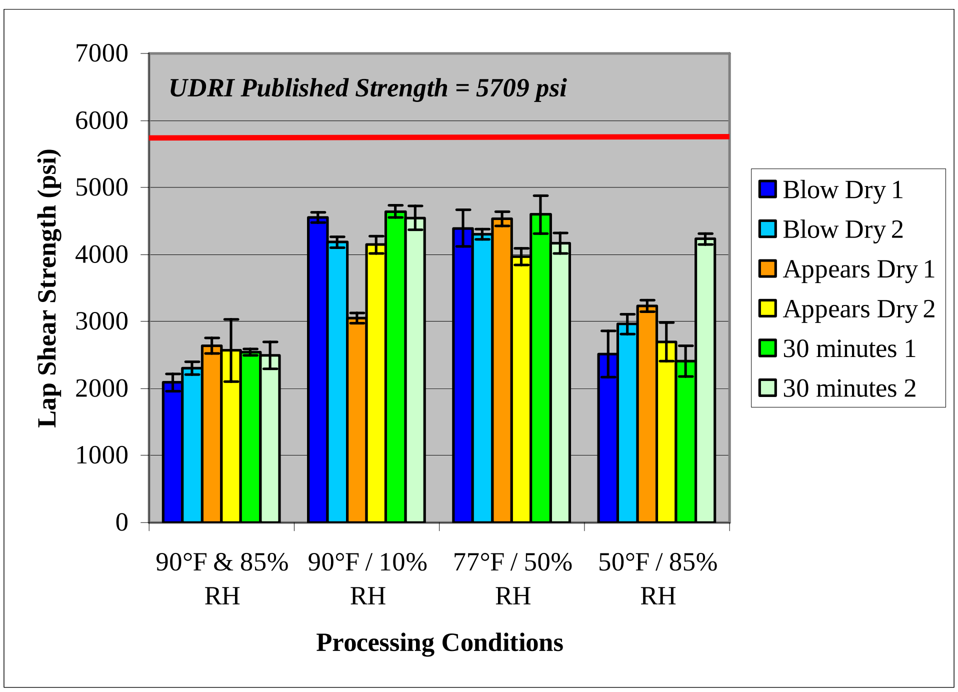


Figure 8: Lap Shear Strength Versus Processing Conditions for GB/SG with BR 6747-1 Cocured with EA 9696 Adhesive

GB/SG with BR 6747-1 Fused then Cocured with EA 9696 Film Adhesive

Wedge test results for GB/SG treated specimens with fused BR 6747-1 cocured with EA 9696 are shown in Table 33. Wedge test results were very consistent, routinely obtaining 95% cohesive failure. Only a single set of specimens exhibited less than 95% cohesive failure. The remaining noncohesive failure occurred at the primer-adhesive interface.

Table 33: Effect of Ambient Conditions and Sol-Gel Dry Method on Bond Durability for Grit-Blast/Sol-Gel with Fused BR 6747-1 Cocured with EA 9696 Adhesive

Ambient Conditions	Sol-Gel Dry Method	Initial (in)	Cumulative Crack Growth (in)							Total (in)	Failure Mode
			1 hr	8 hr	24 hr	7 day	14 day	21 day	28 day		
50°F / 85% RH	Blown dry	1.29	0.08	0.10	0.10	0.18	0.20	0.21	0.22	1.51	95% co
		1.24	0.04	0.04	0.07	0.12	0.13	0.15	0.17	1.42	95% co
	30 minutes	1.31	0.11	0.11	0.12	0.19	0.19	0.22	0.25	1.56	94% co
		1.24	0.04	0.08	0.08	0.15	0.16	0.18	0.18	1.42	95% co
	46 minutes	1.33	0.04	0.06	0.07	0.13	0.14	0.16	0.17	1.51	94% co
		1.30	0.01	0.06	0.06	0.10	0.14	0.14	0.15	1.45	95% co
77°F / 50% RH	Blown dry	1.23	0.06	0.10	0.10	0.15	0.17	0.17	0.17	1.40	95% co
		1.21	0.03	0.07	0.08	0.11	0.13	0.16	0.17	1.38	95% co
	12 minutes	1.29	0.05	0.11	0.13	0.18	0.20	0.20	0.20	1.49	95% co
		1.26	0.02	0.05	0.09	0.13	0.15	0.16	0.18	1.44	95% co
	30 minutes	1.26	0.00	0.02	0.04	0.08	0.11	0.11	0.11	1.37	95% co
		1.23	0.00	0.06	0.06	0.08	0.12	0.15	0.15	1.38	95% co
90°F / 10% RH	Blown dry	1.33	0.02	0.05	0.05	0.10	0.11	0.13	0.13	1.46	95% co
		1.32	0.00	0.00	0.01	0.03	0.07	0.10	0.11	1.43	95% co
	8 minutes	1.31	0.03	0.05	0.07	0.10	0.13	0.14	0.14	1.45	95% co
		1.32	0.00	0.00	0.03	0.07	0.14	0.14	0.14	1.46	95% co
	30 minutes	1.28	0.02	0.04	0.07	0.11	0.11	0.12	0.12	1.40	95% co
		1.26	0.00	0.01	0.04	0.12	0.14	0.15	0.17	1.43	95% co
90°F / 85% RH	Blown dry	1.29	0.06	0.09	0.09	0.15	0.21	0.22	0.22	1.50	95% co
		1.28	0.05	0.08	0.10	0.13	0.15	0.17	0.17	1.46	95% co
	22 minutes	1.19	0.08	0.08	0.08	0.13	0.17	0.18	0.20	1.39	95% co
		1.31	0.06	0.08	0.09	0.12	0.16	0.19	0.19	1.50	95% co
	30 minutes	1.27	0.02	0.02	0.06	0.11	0.16	0.17	0.19	1.46	95% co
		1.29	0.03	0.07	0.09	0.11	0.11	0.14	0.16	1.45	95% co

Lap shear results for GB/SG treated specimens with fused BR 6747-1 cocured with EA 9696 are shown in Table 34. A graphical interpretation of these data is shown in Figure 9. When comparing the results obtained at different temperature/humidity conditions, the lowest strengths were obtained when processing the specimens at 90°F and 85% RH or 50°F and 85% RH. These conditions were the environments with the largest amounts of moisture present in the air and where it proved most difficult to adequately dry the specimens. Conversely, the highest strengths were obtained when processing the specimens at 90°F and 10% RH. This condition proved to be drier and more beneficial for drying the sol-gel. Additionally, the strengths achieved using the primer fusing step

are typically higher and more consistent than the strengths achieved without a primer fusing step (as shown in Table 32 and Figure 8). This trend can be readily seen for the specimens fabricated at 90°F and 85% RH. None of the specimens tested in this effort met the UDRI published strength of 5709 psi.

Table 34: Effect of Ambient Conditions and Sol-Gel Dry Method on Tensile Lap Shear Strength for Grit-Blast/Sol-Gel with Fused BR 6747-1 Cocured with EA 9696 Adhesive

Surface Preparation	Environmental Conditions	Sol-Gel Dry Method	Lap Shear Strength (psi) (Failure Mode)	
			Trial #1	Trial #2
Grit-Blast/Sol-Gel with BR 6747-1 Fused Primer, EA 9696 Adhesive	50°F / 85% RH	Blown dry	4229 (70% co)	4145 (70% co)
	50°F / 85% RH	46 minutes	4147 (70% co)	5025 (80% co)
	50°F / 85% RH	30 minutes	3359 (70% co)	3944 (70% co)
	77°F / 50% RH	Blown dry	3947 (83% co)	4842 (91% co)
	77°F / 50% RH	12 minutes	3667 (85% co)	5055 (94% co)
	77°F / 50% RH	30 minutes	3917 (85% co)	5137 (95% co)
	90°F / 10% RH.	Blown dry	4604 (90% co)	5153 (80% co)
	90°F / 10% RH.	8 minutes	4828 (90% co)	5100 (90% co)
	90°F / 10% RH.	30 minutes	4720 (90% co)	4660 (90% co)
	90°F / 85% RH	Blown dry	3320 (20% co)	3700 (70% co)
	90°F / 85% RH	22 minutes	3823 (80% co)	4392 (85% co)
	90°F / 85% RH	30 minutes	3786 (65% co)	4110 (75% co)

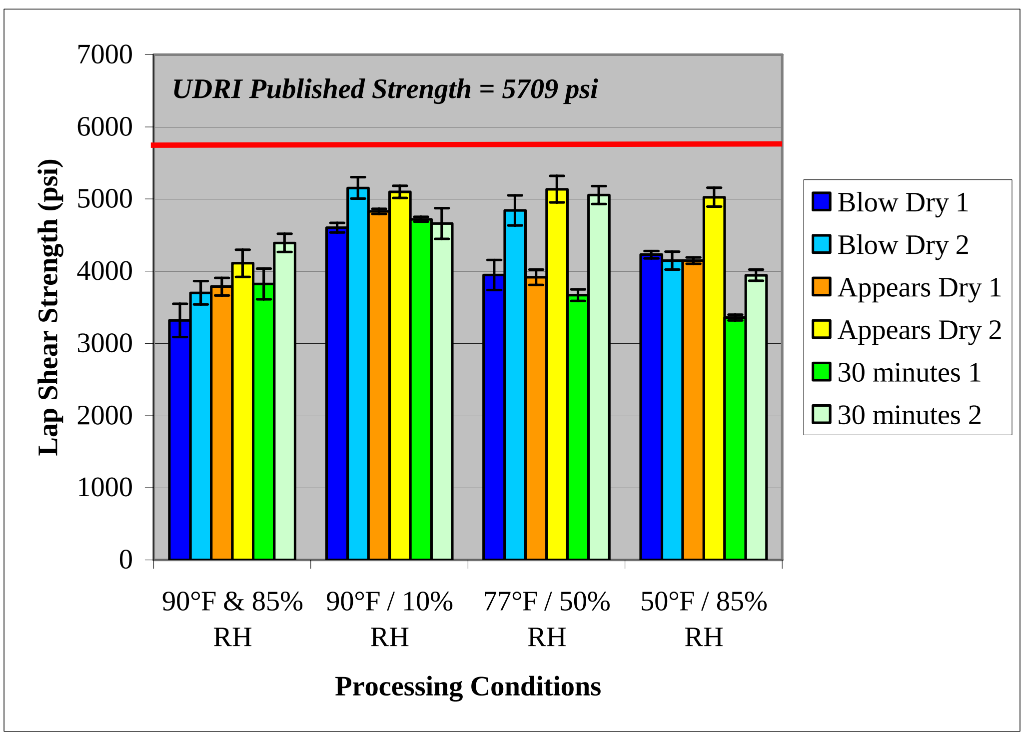


Figure 9: Lap Shear Strength Versus Processing Conditions for GB/SG with Fused BR 6747-1 Cocured with EA 9696 Adhesive

2.4.3 <u>Conclusions</u>

A number of conclusions can be drawn from the evaluation of alternate sol-gel application environments. Foremost, it should be noted the bond performance obtained with sol-gel surface preparations appear to be sensitive to prebond moisture. This can be seen most readily in the lap shear test results for the different surface preparations when comparing strengths achieved from specimens prepared at various ambient temperature and humidity levels. In general, it was more difficult to achieve good lap shear strengths in higher humidity environments. Specific to the is program, specimens fabricated at 50°F and 85% RH as well as 90°F and 85% RH tended to yield lower strengths than specimens fabricated at drier ambient conditions. When comparing the different sol-gel dry steps (blow dry, appears dry, and 30-minute dry at ambient conditions), there did not appear to be much of a difference in wedge test performance or lap shear strength. However, the use of a heat gun to fuse the primer prior to cocuring the primer and adhesive appeared to increase the lap shear strengths of both NP/SG and GB/SG specimens. This step was most likely aiding evaporation of moisture from the bond surface. This moisture could be present due to sol-gel application, BR 6747-1 application or a combination of both. The presence of this prebond moisture during bonding is a likely reason as to why strengths achieved in this effort did not meet previously published UDRI data for EA 9696. Lastly, it should be noted the wedge test did not appear to be sufficient for determining the prebond moisture sensitivity of these surface preparations. Most wedge test specimens routinely failed with less than 0.20 inch of crack growth and >90% cohesive failure modes after 28 days exposure to 120°F and 98% RH. Conversely, results obtained in the lap shear test were very useful in distinguishing between specimens fabricated in different environments.

With the lap shear strength of sol-gel treated specimens being so dependent on the amount of residual moisture on the bond surface, great effort should be taken to remove this moisture from the surface prior to adhesive application and cure. The use of a heat gun has been shown to aid in the evaporation of residual moisture and significantly maximize lap shear strengths for specimens fabricated in moist environments. The use of a heat gun or some other heat source should be implemented in any fielded sol-gel surface preparation to maximize moisture evaporation and improve adhesive properties.

2.5 AC-130 SOL-GEL VERSUS PASA-JELL 105

This project was initiated to perform a comparison of the strength and durability of specimens prepared using AC-130 sol-gel or Pasa-Jell 105. Pasa-Jell 105 is commonly used at the field and depot locations to prepare aluminum for bonded repairs. Pasa-Jell 105 contains strong acids and chromium, therefore it is considered to be a hazardous material. The data generated in this project were meant to provide current Pasa-Jell 105 users with comparable data using AC-130 sol-gel. Several Pasa-Jell 105 and AC-130 sol-gel procedures were investigated. However, a large number of Pasa-Jell 105 process variants exist and were not evaluated.

2.5.1 Test Plan

All specimens in this effort were fabricated from Al 2024-T3 bare sheet stock. Specimens were treated using the surface preparations described in the section below. The general test matrix for this effort is shown in Table 35.

Table 35: Pasa-Jell 105 Test Matrix

Pretreatment	Surface Preparation	Adhesive	Number of Specimens per Test			
			Wedge (120°F)	Wedge (140°F)	Lap Shear (RT)	Peel (RT)
Nylon pad abrasion without bond primer	Pasa-Jell 105-no primer	FM 73M	5	5	5	5
		EA 9696	5	5	5	5
		EA 9394	5	5	5	5
	AC-130-no primer	FM 73M	5	5	5	5
		EA 9696	5	5	5	5
		EA 9394	5	5	5	5
Nylon pad abrasion with bond primer	Pasa-Jell 105 & BR 127	FM 73M	5	5	5	5
		EA 9696	5	5	5	5
	Pasa-Jell 105 & BR 6747-1	FM 73M	5	5	5	5
		EA 9696	5	5	5	5
	AC-130 & BR 6747-1	FM 73M	5	5	5	5
		EA 9696	5	5	5	5
Grit-blast with bond primer	Pasa-Jell 105 & BR 127	FM 73M	5	5	5	5
		EA 9696	5	5	5	5
	Pasa-Jell 105 & BR 6747-1	FM 73M	5	5	5	5
		EA 9696	5	5	5	5
	AC-130 & BR 6747-1	FM 73M	5	5	5	5
		EA 9696	5	5	5	5

Surface Preparation Procedures

Nylon Pad Abrasion without Bond Primer - Adherends prepared using the nylon pad abrasion process without bond primer were initially degreased using acetone-soaked, lint-free wipes. The bond surfaces were abraded using a MED Scotch-Brite Roloc pad and degreased again with an acetone-soaked, lint-free wipe. Pasa-Jell 105 was applied using an acid brush and agitated on the surface for 20 minutes. This 20-minute Pasa-Jell 105 exposure was a compromise between a 30-minute exposure recommended by NAVAIR and a 10-15 minute exposure recommended by MIL-HDBK-337[21]. The Pasa-Jell 105 was removed from the adherend using a lint-free wipe soaked in deionized water. Litmus paper was used to verify the acid had been removed from the surface. When using AC-130, the sol-gel was applied with an acid brush, keeping the surface wet for three minutes. Sol-gel treated surfaces were dried at ambient temperature (70°F) for 30 minutes prior to adhesive application.

Nylon Pad Abrasion with Bond Primer - Adherends prepared using the nylon pad abrasion process with bond primer were initially degreased using acetone-soaked, lint-free wipes. The bond surfaces were abraded using a MED Scotch-Brite Roloc pad and degreased again with an acetone-soaked, lint-free wipe. Pasa-Jell 105 was applied using an acid brush and agitated on the surface for 20 minutes. The Pasa-Jell 105 was removed from the adherend using a lint-free wipe soaked in deionized water. Litmus paper was used to verify the acid had been removed from the surface. When using AC-130, the sol-gel was applied with an acid brush keeping the surface wet for 3 minutes. Sol-gel treated surfaces were dried at ambient laboratory conditions (70°F and 40% RH) for 30 minutes prior to primer application.

Pasa-Jell 105 treated specimens were primed with either BR 127 or BR 6747-1 primer. BR 127 was applied using a Binks 105 spray gun, dried at ambient conditions for 30 minutes, and cured for 60 minutes at 250°F in an air-circulating oven. BR 6747-1 was applied to Pasa-Jell 105-treated surfaces with a foam brush using the process specified in Section 2.1.1. The primer was dried at ambient conditions for 30 minutes and cocured with the adhesive at 250°F. Sol-gel treated surfaces were primed with BR 6747-1 using the process specified in Section 2.1.1. The primer was dried at ambient conditions for 30 minutes and cocured with the adhesive at 250°F.

Grit-Blast Deoxidation with Bond Primer - Adherends prepared using the grit-blast deoxidation process with bond primer were initially degreased using acetone-soaked, lint-free wipes. The bond surfaces were abraded using a VFN Scotch-Brite Roloc pad and degreased again with an acetone-soaked, lint-free wipe. Bond surfaces were grit-blasted using 50 μm Al_2O_3 and blown clean with 35 psi N_2. Pasa-Jell 105 was applied using an acid brush and agitated on the surface for 20 minutes. The Pasa-Jell 105 was removed from the adherend using a lint-free wipe soaked in deionized water. Litmus paper was used to verify the acid had been removed from the surface. When using AC-130, the sol-gel was applied with an acid brush keeping the surface wet for 3 minutes then dried at ambient conditions for 30 minutes prior to primer application.

Pasa-Jell 105 treated specimens were primed with either BR 127 or BR 6747-1 primers. BR 127 was applied using a Binks 105 spray gun, dried at ambient conditions for 30 minutes, and cured for 60 minutes at 250°F in an air-circulating oven. BR 6747-1 was applied to Pasa-Jell 105-treated surfaces with a foam brush using the process specified in Section 2.1.1. The primer was dried at ambient conditions for 30 minutes and cocured with the adhesive at 250°F. Sol-gel treated surfaces were primed with BR 6747-1 using the process specified in Section 2.1.1. The primer was dried at ambient conditions for 30 minutes and cocured with the adhesive at 250°F.

Specimen Bonding

Specimens were bonded using either Cytec FM 73M (0.06 psf) or Hysol EA 9396 (0.06 psf) epoxy film adhesives, or Hysol EA 9394 two-part epoxy paste adhesive. Specimens bonded with FM 73 and EA 9696 were cured for 1 hour at 250°F and 35 psi in a portable autoclave. Specimens bonded with EA 9394 included a 5 mil (0.005 in) random mat polyester scrim cloth for bondline control and were cured for 60 minutes at 150°F and 35 psi in a portable autoclave.

Specimen Machining

Tensile lap shear panels and wedge test panels were machined into 1-inch wide specimens using a gang-cutting mill. Floating roller peel panels were machined into ½-inch wide specimens using a metal foot shear. Bondline thicknesses were measured using an optical microscope.

Specimen Testing

Tensile lap shear specimens were tested according to ASTM D 1002, floating roller peel specimens were tested according to ASTM D 3176, and wedge tests were conducted according to ASTM D 3762. Wedge test specimens were tested at one of two conditions: (1) 120°F and 98% RH or (2) 140°F and 98% RH. Crack growth was monitored for wedge test specimens after 1 hour, 8 hours, 24 hours, 7 days, 14 days, 21 days, and 28 days. Failure modes were determined for all specimens once testing was complete and identified as a percentage of cohesive failure.

2.5.2 Test Results

Nylon Pad Abrasion without Bond Primer Test Results

Tensile lap shear and floating roller peel test results for nylon pad abraded specimens without primer are shown in Table 36. In general, specimens prepared with AC-130 exhibited similar strengths and better failure modes than specimens treated with Pasa-Jell 105 when using a nylon pad abrasion without use of a bond primer.

Table 36: Tensile Lap Shear and Floating Roller Peel Test Results for Nylon Pad Abraded Specimens without Bond Primer

Surface Preparation	Adhesive	RT Bond Strength	
		Lap Shear (psi) [failure mode]	Peel (pli) [failure mode]
Pasa-Jell 105 - no primer	FM 73M	4750 [91% co]	71.5 [71% co]
	EA 9696	5149 [83% co]	66.5 [60% co]
	EA 9394	4090 [10% co]	3.7 [0% co]
AC-130 - no primer	FM 73M	5123 [100% co]	72.9 [100% co]
	EA 9696	5059 [100% co]	65.0 [100% co]
	EA 9394	3937 [64% co]	15.8 [2% co]

Wedge test results after 28 days exposure to 120°F and 98% RH are shown in Table 37. AC-130 specimens exhibited similar crack growth and failure modes when compared to Pasa-Jell 105 specimens, except specimens bonded with EA 9394 paste adhesive. It should be noted, however, use of neither AC-130 nor Pasa-Jell 105 with the nylon pad abrasion (no primer) process provided acceptable failure modes in the wedge test with any of the adhesives. All specimens exhibited very low amounts of cohesive failure; none exhibited more than 65% cohesive failure.

Table 37: Wedge Test Results (120°F & 98% RH) for Nylon Pad Abraded Specimens without Bond Primer

Surface Preparation	Adhesive	Initial (in)	Cumulative Crack Growth (in)							Failure Mode (% cohesive)
			1 hr	8 hr	24 hrs	7 days	14 days	21 days	28 days	
Pasa-Jell 105	FM 73M	1.24	0.15	0.69	1.38	2.14	2.28	2.28	2.30	0% co
	EA 9696	1.29	0.10	0.11	0.15	0.27	0.29	0.32	0.32	34% co
	EA 9394	1.72	0.32	0.41	0.46	0.49	0.53	0.53	0.53	65% co
AC-130	FM 73M	1.22	0.04	0.08	0.13	0.50	0.59	0.65	0.69	0% co
	EA 9696	1.36	0.00	0.07	0.12	0.20	0.26	0.27	0.28	44% co
	EA 9394	1.78	0.03	0.19	0.56	0.78	0.80	0.80	0.80	16% co

Wedge test results after 28 days exposure to 140°F and 98% RH are shown in Table 38. AC-130 specimens exhibited similar crack growth and failure modes when compared to Pasa-Jell 105 specimens, except specimens bonded with EA 9394 paste adhesive. Pasa-Jell 105 appeared to provide better bond durability (less crack growth and higher percentage of cohesive failure) when using EA 9394 paste adhesive. As with the wedge test specimens exposed to 120°F and 98% RH, use of AC-130 or Pasa-Jell 105 with the nylon pad abrasion (no primer) process did not provide acceptable failure modes in the wedge test with EA 9696 or FM 73M. All specimens exhibited very low amounts of cohesive failure and, other than the Pasa-Jell 105 specimens bonded with EA 9394, none yielded more than 22% cohesive failure.

Table 38: Wedge Test Results (140°F & 98% RH) for Nylon Pad Abraded Specimens without Bond Primer

Surface Preparation	Adhesive	Initial (in)	Cumulative Crack Growth (in)							Failure Mode (% cohesive)
			1 hr	8 hr	24 hrs	7 days	14 days	21 days	28 days	
Pasa-Jell 105	FM 73M	1.31	0.10	0.75	1.29	1.59	1.59	1.59	1.59	0% co
	EA 9696	1.39	0.07	0.14	0.21	0.29	0.29	0.29	0.30	22% co
	EA 9394	1.76	0.04	0.09	0.11	0.13	0.18	0.25	0.26	93% co
AC-130	FM 73M	1.22	0.03	0.09	0.10	0.41	0.48	0.53	0.55	0% co
	EA 9696	1.28	0.01	0.06	0.11	0.18	0.19	0.21	0.22	22% co
	EA 9394	1.75	0.15	0.23	0.36	0.51	0.53	0.54	0.54	54% co

The test data presented in this section illustrate that specimens treated with AC-130 sol-gel provide similar bond strength and durability to specimens treated with Pasa-Jell 105 when employing a nylon pad abrasion pretreatment without use of a bond primer.

Nylon Pad Abrasion with Bond Primer

Tensile lap shear and floating roller peel test results for nylon pad abraded specimens with primer are shown in Table 39. There was not much difference noticed in the lap shear strengths for specimens treated with Pasa-Jell 105 or AC-130 sol-gel. However, the Pasa-Jell 105 specimens primed with BR 127 possessed peel strengths that were less than half of strengths achieved with Pasa-Jell 105 and AC-130 when primed with BR 6747-1. There was also a corresponding loss in the amount of cohesive failure observed after the test. The noncohesive failure occurred between the primer and adherend. There was not much difference in the lap shear or peel strengths when comparing specimens treated with Pasa-Jell 105 to specimens treated with AC-130 when primed with BR 6747-1 in both cases. Good failure modes were also observed in lap shear and peel tests when using BR 6747-1 with both AC-130 and Pasa-Jell 105.

Table 39: Tensile Lap Shear and Floating Roller Peel Test Results for Nylon Pad Abraded Specimens with Bond Primer

Surface Preparation	Adhesive	RT Bond Strength	
		Lap Shear (psi) [failure mode]	Peel (pli) [failure mode]
Pasa-Jell 105 & BR 127	FM 73M	5416 [100% co]	34.9 [58% co]
	EA 9696	6009 [95% co]	25.7 [52% co]
Pasa-Jell 105 & BR 6747-1	FM 73M	5357 [97% co]	80.0 [100% co]
	EA 9696	5491 [99% co]	69.9 [86% co]
AC-130 & BR 6747-1	FM 73M	5377 [99% co]	72.8 [100% co]
	EA 9696	5478 [99% co]	64.4 [100% co]

Wedge test results (120°F and 98% RH) for nylon pad abraded specimens with bond primer are shown in Table 40. All noncohesive failure occurred between the primer and substrate. Pasa-Jell 105, when combined with BR 127, provides poor bond durability as exhibited by the excessive crack growth and poor failure modes. When compared to Pasa-Jell 105/BR 127 results, use of Pasa-Jell 105 with BR 6747-1 improves the wedge test performance when using EA 9696. Use of AC-130 sol-gel does not improve wedge test results when using FM 73M adhesive, but drastically improves wedge test performance when used in with EA 9696 adhesive.

Table 40: Wedge Test Results (120°F & 98% RH) for Nylon Pad Abraded Specimens with Bond Primer

Surface Preparation	Adhesive	Initial (in)	Cumulative Crack Growth (in)							Failure Mode (% cohesive)
			1 hr	8 hr	24 hrs	7 days	14 days	21 days	28 days	
Pasa-Jell 105 & BR 127	FM 73M	1.26	0.51	0.62	0.62	0.62	0.64	0.66	0.67	22% co
	EA 9696	1.38	0.95	1.04	1.05	1.07	1.07	1.09	1.09	6% co
Pasa-Jell 105 & BR 6747-1	FM 73M	1.32	0.03	0.06	0.07	0.12	0.19	0.31	0.38	0% co
	EA 9696	1.36	0.04	0.05	0.12	0.18	0.21	0.28	0.28	70% co
AC-130 & BR 6747-1	FM 73M	1.32	0.05	0.08	0.08	0.23	0.51	0.61	0.65	0% co
	EA 9696	1.35	0.00	0.04	0.04	0.14	0.17	0.19	0.20	95% co

Wedge test results after 28 days exposure to 140°F and 98% RH are shown in Table 41. All noncohesive failure occurred between the primer and substrate. Similar trends were noticed in the 140°F wedge testing as observed in the 120°F. Specimens bonded with FM 73M did not perform well with any of the surface pretreatments. Pasa-Jell 105-treated specimens performed better when used with BR 6747-1 and EA 9696 adhesive. AC-130-treated specimens also performed well when used with EA 9696, exhibiting better failure modes than Pasa-Jell 105/ BR 6747-1 specimens.

Table 41: Wedge Test Results (140°F & 98% RH) for Nylon Pad Abraded Specimens with Bond Primer

Surface Preparation	Adhesive	Initial (in)	Cumulative Crack Growth (in)							Failure Mode (% cohesive)
			1 hr	8 hr	24 hrs	7 days	14 days	21 days	28 days	
Pasa-Jell 105 & BR 127	FM 73M	1.23	0.35	0.37	0.37	0.45	0.52	0.54	0.58	0% co
	EA 9696	1.32	0.56	0.59	0.60	0.61	0.63	0.65	0.67	12% co
Pasa-Jell 105 & BR 6747-1	FM 73M	1.25	0.10	0.10	0.12	0.35	0.47	0.53	0.57	0% co
	EA 9696	1.45	0.05	0.08	0.11	0.15	0.20	0.21	0.23	65% co
AC-130 & BR 6747-1	FM 73M	1.26	0.07	0.09	0.09	0.24	0.34	0.39	0.44	0% co
	EA 9696	1.37	0.03	0.09	0.13	0.18	0.20	0.25	0.25	83% co

The test data presented in this section illustrate specimens treated with AC-130 sol-gel provide similar bond strength and durability to specimens treated with Pasa-Jell 105 when employing a nylon pad abrasion pretreatment with use of a BR 6747-1 bond primer. Pasa-Jell 105 tended to exhibit better bond strength and durability when used with BR 6747-1 versus BR 127. Lastly, better wedge test results were obtained when using EA 9696 versus FM 73M. In fact, wedge test specimens bonded with FM 73M exhibited poor failure modes and excessive crack growth.

Grit-Blast Deoxidation with Bond Primer

Tensile lap shear and floating roller peel test results for grit-blasted specimens with primer are shown in Table 42. There was not much difference noticed in the lap shear strengths for specimens treated with Pasa-Jell 105 or AC-130 sol-gel. However, the Pasa-Jell 105 specimens primed with BR 127 possessed peel strengths less than half of strengths achieved with Pasa-Jell 105 and AC-130 when primed with BR 6747-1. There was not much difference in the lap shear or peel strengths when comparing specimens treated with Pasa-Jell 105 to specimens treated with AC-130 when primed with BR 6747-1 in both cases. Although, higher peel strengths were achieved with FM 73M when compared to similar specimens bonded with EA 9696. Good failure modes were observed in lap shear and peel tests with both AC-130 and Pasa-Jell 105.

Table 42: Tensile Lap Shear and Floating Roller Peel Test Results for Grit-Blasted Specimens with Bond Primer

Surface Preparation	Adhesive	RT Bond Strength	
		Lap Shear (psi) [failure mode]	Peel (pli) [failure mode]
Pasa-Jell 105 & BR 127	FM 73M	5536 [97% co]	37.8 [100% co]
	EA 9696	5762 [100% co]	27.5 [100% co]
Pasa-Jell 105 & BR 6747-1	FM 73M	4912 [100% co]	76.5 [83% co]
	EA 9696	5279 [95% co]	59.4 [100% co]
AC-130 & BR 6747-1	FM 73M	5226 [99% co]	82.7 [100% co]
	EA 9696	5158 [100% co]	52.9 [100% co]

Wedge test results obtained after aging grit-blast deoxidized specimens at 120°F and 98% RH are shown in Table 43. All noncohesive failure occurred between the primer and substrate. Similar to previous wedge test results, specimens bonded with EA 9696 exhibited shorter crack growth and better failure modes than similar specimens bonded with FM 73M. There was not much difference between crack growth and failure modes of specimens bonded with EA 9696 adhesive when comparing the use of Pasa-Jell 105 and AC-130. However, Pasa-Jell 105 specimens primed with BR 6747-1 and bonded with FM 73M exhibit excessive crack growth and complete interfacial failure between the primer and substrate.

Table 43: Wedge Test Results (120°F & 98% RH) for Grit-Blasted Specimens with Bond Primer

Surface Preparation	Adhesive	Initial (in)	Cumulative Crack Growth (in)							Failure Mode (% cohesive)
			1 hr	8 hr	24 hrs	7 days	14 days	21 days	28 days	
Pasa-Jell 105 & BR 127	FM 73M	1.24	0.06	0.07	0.07	0.08	0.08	0.09	0.09	91% co
	EA 9696	1.39	0.03	0.07	0.09	0.13	0.18	0.19	0.21	99% co
Pasa-Jell 105 & BR 6747-1	FM 73M	1.30	0.08	0.17	0.31	0.61	0.75	0.78	0.86	0% co
	EA 9696	1.34	0.03	0.08	0.08	0.15	0.16	0.18	0.20	93% co
AC-130 & BR 6747-1	FM 73M	1.23	0.03	0.03	0.03	0.07	0.13	0.20	0.22	39% co
	EA 9696	1.36	0.00	0.03	0.04	0.12	0.14	0.16	0.17	100% co

Wedge test results obtained after aging at 140°F and 98% RH are shown in Table 44. All noncohesive failure occurred between the primer and substrate. Results obtained in the 140°F wedge tests were very similar to the results obtained in the 120°F wedge tests for grit-blasted surfaces. Specimens bonded with EA 9696 exhibited better failure modes than similar specimens bonded with FM 73M. There was not much difference between crack growth and failure modes of specimens bonded with EA 9696 adhesive when comparing the use of Pasa-Jell 105 and AC-130. However, Pasa-Jell 105 specimens primed with BR 6747-1 and bonded with FM 73M exhibit excessive crack growth and complete interfacial failure between the primer and substrate.

Table 44: Wedge Test Results (140°F & 98% RH) for Grit-Blasted Specimens with Bond Primer

Surface Preparation	Adhesive	Initial (in)	Cumulative Crack Growth (in)							Failure Mode (% cohesive)
			1 hr	8 hr	24 hrs	7 days	14 days	21 days	28 days	
Pasa-Jell 105 & BR 127	FM 73M	1.33	0.02	0.08	0.08	0.12	0.19	0.23	0.24	59% co*
	EA 9696	1.32	0.06	0.14	0.14	0.22	0.23	0.26	0.27	88% co
Pasa-Jell 105 & BR 6747-1	FM 73M	1.30	0.05	0.05	0.08	0.37	0.46	0.50	0.54	0% co
	EA 9696	1.29	0.07	0.11	0.13	0.19	0.21	0.23	0.24	89% co
AC-130 & BR 6747-1	FM 73M	1.26	0.05	0.08	0.09	0.15	0.18	0.26	0.26	36% co
	EA 9696	1.40	0.01	0.09	0.09	0.17	0.21	0.24	0.26	92% co*

* noncohesive failure occurred between adhesive and primer

The test data presented in this section illustrate specimens treated with AC-130 sol-gel provide similar bond strength and durability to specimens treated with Pasa-Jell 105 when employing a grit-blast deoxidation. Better wedge test results were obtained when using EA 9696 versus FM 73M, especially when using the BR 6747-1 primer. Wedge test specimens primed with

BR 6747-1 and bonded with FM 73M exhibited poor failure modes and excessive crack growth. This was unexpected since Cytec manufactures both FM 73M and BR 6747-1.

2.5.3 Conclusions

AC-130 sol-gel appears to perform as well as, or better than, Pasa-Jell 105 when used with the various pretreatments, adhesives, and bond primers used in this project. Pasa-Jell 105 bonds were extremely susceptible to moisture (i.e. wedge test data) when performed without grit-blasting. Pasa-Jell 105 bonds also exhibited better failure modes when used with BR 6747-1 bond primer rather than BR 127. Specimens bonded with FM 73M exhibited poor failure modes as compared to similar specimens bonded with EA 9696, especially when treating the adherends with AC-130 sol-gel and priming with BR 6747-1. This suggests there is a concern using FM 73M with sol-gel surface preparations and BR 6747-1 primer.

2.6 AC-130 SOL-GEL VERSUS LAB MIXED BOEGEL-EPII SOL-GEL

On several instances throughout this program, the performance of the sol-gel surface preparations proved to be inferior to that previously observed during the Strategic Environmental Research and Development Program (SERDP) PP-1113 project[22]. Several variables were the possible cause of this phenomenon, but one obvious difference was identified. During most of the SERDP program, bonded specimens were treated using Boegel-EPII mixed fresh from raw chemicals. However, during most of the ESTCP PP-0204 effort, specimens were treated with AC-130 sol-gel kits manufactured by Advanced Chemistry and Technology (AC Tech) and acquired through the General Services Administration (GSA). Each 100 mL kit was ordered through the GSA website using a national stock number (NSN 6850-01-505-8844). One possible cause of the erratic test results using the sol-gel surface preparations was hypothesized to be due to use of the AC-130 kits.

2.6.1 Test Plan

A small test program was conducted to investigate if any difference in bond performance existed between AC-130 kits (commercially available Boegel-EPII) and laboratory-mixed Boegel-EPII from fresh chemicals. In order to investigate this phenomenon, several bonding variables were evaluated, including:

- grit-blast versus nylon pad abrasion,
- AC-130 sol-gel versus fresh laboratory Boegel-EPII,
- spray application versus brush application for bond primer,
- precure of bond primer versus cocuring the adhesive and bond primer, and
- 3M Company AF 163-2M adhesive versus Henkel's Hysol EA 9696 adhesive.

The test matrix for this investigation is shown in Table 45. The matrix was executed twice so there were two wedge test panels fabricated for each test condition.

Table 45: AC-130 Sol-Gel Kit versus Fresh Laboratory

Pretreatment	Sol-Gel	Primer Application	Primer Cure	Adhesive	Wedge Test 120°F & 98% RH
Grit-Blast	AC-130	Spray	Precure	AF 163-2M	10 specimens
Grit-Blast	Fresh Mixed	Spray	Precure	AF 163-2M	10 specimens
Grit-Blast	AC-130	Spray	Cocure	AF 163-2M	10 specimens
Grit-Blast	Fresh Mixed	Spray	Cocure	AF 163-2M	10 specimens
Grit-Blast	AC-130	Brush	Cocure	AF 163-2M	10 specimens
Grit-Blast	Fresh Mixed	Brush	Cocure	AF 163-2M	10 specimens
Grit-Blast	AC-130	Spray	Precure	EA 9696	10 specimens
Grit-Blast	Fresh Mixed	Spray	Precure	EA 9696	10 specimens
Grit-Blast	AC-130	Spray	Cocure	EA 9696	10 specimens
Grit-Blast	Fresh Mixed	Spray	Cocure	EA 9696	10 specimens
Grit-Blast	AC-130	Brush	Cocure	EA 9696	10 specimens
Grit-Blast	Fresh Mixed	Brush	Cocure	EA 9696	10 specimens
Nylon-Pad	AC-130	Spray	Precure	AF 163-2M	10 specimens
Nylon-Pad	Fresh Mixed	Spray	Precure	AF 163-2M	10 specimens
Nylon-Pad	AC-130	Spray	Cocure	AF 163-2M	10 specimens
Nylon-Pad	Fresh Mixed	Spray	Cocure	AF 163-2M	10 specimens
Nylon-Pad	AC-130	Brush	Cocure	AF 163-2M	10 specimens
Nylon-Pad	Fresh Mixed	Brush	Cocure	AF 163-2M	10 specimens
Nylon-Pad	AC-130	Spray	Precure	EA 9696	10 specimens
Nylon-Pad	Fresh Mixed	Spray	Precure	EA 9696	10 specimens
Nylon-Pad	AC-130	Spray	Cocure	EA 9696	10 specimens
Nylon-Pad	Fresh Mixed	Spray	Cocure	EA 9696	10 specimens
Nylon-Pad	AC-130	Brush	Cocure	EA 9696	10 specimens
Nylon-Pad	Fresh Mixed	Brush	Cocure	EA 9696	10 specimens

Specimen Fabrication

The test matrix consisted of wedge tests per ASTM D 3762 using bare Al 2024-T3 adherends. Grit-blasted specimens were prepared by initially degreasing the specimens using acetone-soaked, lint-free wipes until the surfaces were clean. The bond surfaces were abraded using

MED Scotch-Brite Roloc pads on a 20,000-RPM grinder. The surfaces were cleaned with acetone-soaked, lint-free wipes again then grit-basted with 50μm Al_2O_3. The grit-blasted surfaces were blown with 35-psi clean air in order to remove any residual grit on the surface. Sol-gel was applied with an acid brush so the surfaces were wetted for a minimum of three minutes. Adherends were then placed in a vertical rack to drain and dry for 30 minutes at ambient laboratory conditions (70°F and 40% RH). Once dry, Cytec BR 6747-1 primer was applied using either a Binks 105 spray gun or a foam brush (per section 2.1.1) to a thickness of 0.1-0.3 mil (0.0001-0.0003 inch). The primed panels were dried at ambient conditions for 30 minutes. Once dried, the primed panels were either precured in an air-circulating oven for 1 hour at 250°F or cocured with the adhesive after fusing the primer in an air-circulating oven for 10 minutes at 200°F. All specimens were bonded using either AF 163-2M (0.06 psf) or EA 9696 (0.06 psf) in a portable autoclave for 1 hour at 250°F and 35 psi.

All wedge test specimens were machined to one-inch wide using a gang-cutting mill. The ends of the specimens were polished with a belt sander to enable measurement of bondline thicknesses with the use of an optical microscope.

Crack Extension Testing (ASTM D 3762 Wedge Test)

Stainless steel (301) wedges were inserted into the wedge test specimens using a hammer. The initial crack lengths were measured and recorded for each specimen. Specimens were then exposed to 120°F and 98% RH for 28 days. Crack growth was recorded after 1 hour, 8 hours, 24 hours, 7 days, 14 days, 21 days, and 28 days. Once the 28-day measurement was recorded, the stainless steel wedges were removed from the specimens and the specimens were opened using a vice and a pair of vice grips. The failure modes of the specimens were recorded as the percentage of test area exhibiting cohesive (within the adhesive) failure.

2.6.2 Test Results

Two runs of the test matrix were separately performed so that there would be two independent sets of data to evaluate. Wedge test results from the first round are shown in Table 46. Using only the crack growth after 28 days as a comparison, there did not appear to be much difference in the data. All of the data sets exhibited crack growth of 0.20 inch or less except for two sets.

Both of the sets exhibiting greater than 0.20 inch crack growth were abraded with nylon pads, treated with AC-130 sol-gel, and had the primer and adhesive cocured during processing.

There were a number of differences noticed when comparing failure modes. In general, the following trends were noticed:

- Four sets of specimens exhibited >95% cohesive failure mode indicating the surface preparation was adequate. All of these specimens were grit-blasted.
- Twelve sets of specimens exhibited failure between the adhesive and primer. This was not deemed to be a surface preparation failure since the failure did not occur between the primer and metal. No trends were noticed with the processing steps causing this type of failure. The phenomenon appeared to be random.
- Eight sets of specimens failed with a significant portion of the failure occurring between the primer and metal, indicating an inadequate surface preparation. The following trends were noticed:
    - 7/8 of the sets were bonded using EA 9696 adhesive
    - 7/8 of the sets had primer cocured with the adhesive
    - 6/8 of the sets were abraded with nylon pads

Table 46: Wedge Test Results from the First Round

Pretreatment	Adhesive	Sol-gel	Primer Method	Primer Cure	Initial (in)	28 Day Growth (in)	Failure Mode (% co)
Grit-blast	AF 163-2M	AC-130	Spray	Precure	1.13	0.13	64% *
		Lab mix		Precure	1.15	0.13	60% *
		AC-130		Cocure	1.11	0.16	95%
		Lab mix		Cocure	1.11	0.15	95%
		AC-130	Brush	Cocure	1.14	0.16	79% *
		Lab mix	Brush	Cocure	1.14	0.19	61% *
	EA 9696	AC-130	Spray	Precure	1.21	0.14	100%
		Lab mix		Precure	1.21	0.12	100%
		AC-130		Cocure	1.23	0.19	76% **
		Lab mix		Cocure	1.20	0.18	85% *
		AC-130	Brush	Cocure	1.23	0.19	28% **
		Lab mix	Brush	Cocure	1.23	0.12	74% *
Nylon pad	AF 163-2M	AC-130	Spray	Precure	1.15	0.14	73% *
		Lab mix		Precure	1.07	0.10	79% *
		AC-130		Cocure	1.17	0.22	79% **
		Lab mix		Cocure	1.19	0.19	86% *
		AC-130	Brush	Cocure	1.12	0.14	76% *
		Lab mix	Brush	Cocure	1.14	0.16	73% *
	EA 9696	AC-130	Spray	Precure	1.21	0.13	80% *
		Lab mix		Precure	1.21	0.13	90% **
		AC-130		Cocure	1.32	0.14	46% **
		Lab mix		Cocure	1.23	0.18	58% **
		AC-130	Brush	Cocure	1.22	0.35	12% **
		Lab mix	Brush	Cocure	1.17	0.08	74% **

* Noncohesive failure occurred between primer and adhesive

** Noncohesive failure occurred between primer and metal

Wedge test results for the second round are shown in Table 47. As witnessed in the first round, the specimens exhibited relatively short crack growth after 28 days exposure to 120°F and 98% RH. Only three specimen sets exhibited crack growth in excess of 0.20 inch. All three of those specimen sets were abraded with nylon pads and were fabricated using a single cocure cycle for the primer and adhesive.

Table 47: Wedge Test Results from the Second Round

Pretreatment	Adhesive	Sol-gel	Primer Method	Primer Cure	Initial (in)	28 Day Growth (in)	Failure Mode (% co)
Grit-blast	AF 163-2M	AC-130	Spray	Precure	1.18	0.13	93% *
		Lab mix		Precure	1.26	0.17	94% *
		AC-130		Cocure	1.11	0.17	95%
		Lab mix		Cocure	1.15	0.16	92% *
		AC-130	Brush	Cocure	1.21	0.17	74% *
		Lab mix	Brush	Cocure	1.26	0.19	73% *
	EA 9696	AC-130	Spray	Precure	1.14	0.16	99%
		Lab mix		Precure	1.13	0.11	100%
		AC-130		Cocure	1.12	0.18	82% *
		Lab mix		Cocure	1.06	0.14	100%
		AC-130	Brush	Cocure	1.20	0.19	46% *
		Lab mix	Brush	Cocure	1.19	0.19	36% *
Nylon pad	AF 163-2M	AC-130	Spray	Precure	1.12	0.14	94%
		Lab mix		Precure	1.15	0.18	79% **
		AC-130		Cocure	1.18	0.24	59% **
		Lab mix		Cocure	1.25	0.42	0% **
		AC-130	Brush	Cocure	1.17	0.19	80% **
		Lab mix	Brush	Cocure	1.13	0.18	72% **
	EA 9696	AC-130	Spray	Precure	1.11	0.13	62% **
		Lab mix		Precure	1.07	0.15	84% **
		AC-130		Cocure	1.09	0.16	50% **
		Lab mix		Cocure	1.12	0.15	33% **
		AC-130	Brush	Cocure	1.18	0.24	36% **
		Lab mix	Brush	Cocure	1.14	0.22	18% **

*   Noncohesive failure occurred between primer and adhesive
** Noncohesive failure occurred between primer and metal

As with the first round of wedge testing, a larger variation in results was witnessed when comparing failure modes from the wedge testing in the second round. The following trends were noticed when comparing failure modes from specimens tested in the second round:

- Four sets of wedge test specimens exhibited >95% cohesive failure mode, indicating the surface preparation was adequate. All of these specimens were grit-blasted.
- Eight sets of wedge test specimens exhibited failure between the adhesive and primer. This was not deemed to be a surface preparation failure since the failure did not occur between the primer and metal. All of these specimens were grit-blasted.
- All of the grit-blasted wedge test specimens possessed either cohesive failures or failure between the adhesive and primer.

- One set of wedge test specimens exhibited complete adhesive failure between the primer and substrate. That set was abraded with a nylon pad, treated using lab mix, and cocured with AF 163-2M.
- Eleven sets of wedge test specimens failed with a significant portion of the failure occurring between the primer and metal, thus indicating an inadequate surface preparation. The only apparent trend was all 11 sets of the specimens were abraded with a nylon pad.

2.6.3 Conclusions

The only common trend between both rounds of wedge testing concerns the use of nylon pad abrasion versus grit-blasting. Grit-blasted specimens exhibited better failure modes than failure modes achieved with nylon pad abrasion. This trend was noticeable in the first round of wedge testing and even more pronounced in the second round of wedge testing. In fact, it is possible that the use of grit-blasting versus nylon pad abrasion was so significant, it overrode all other evaluated factors in the second round of testing. Even still, several trends from the first round of testing showed the combined use of nylon pad abrasion, bonding with EA 9696 film adhesive, and cocuring EA 9696 with BR 6747-1 primer caused reduced amounts of cohesive failure in wedge test specimens aged for 28 days at 120°F. This trend is suspicious since results from Section 2.1 showed that this same combination produced excellent wedge test results. Possible causes for the sudden change could be primer batch, adhesive batch, or another uncontrolled variable that has yet to be identified. Lastly, both rounds of testing did not identify any trends with using AC-130 sol-gel versus freshly mixed sol-gel from chemical stock.

3 TASK #2: GENERATION AND DOCUMENTATION OF BONDED REPAIR MATERIALS PROPERTY DATA

On-aircraft processing techniques limit the ability to attain tight temperature tolerances and process these systems with positive pressure. Instead, on-aircraft repairs are typically accomplished using vacuum bag pressure and curing over a wide range of temperatures due to uneven aircraft substructure. Processing composites in this manner can result in poor laminate quality, reduced physical properties (T_g, degree of cure, etc), and reduced mechanical properties (strength, modulus, strain to failure, etc.). These reduced properties must be considered when designing composite repairs. Typically, engineers use material design allowables provided by composite manufacturers to design composite repairs. These design allowables are typically generated using optimized processing techniques, including positive pressure and controlled heating. Engineers account for loss of properties due to on-aircraft processing by applying an estimated "knockdown factor." These estimated knockdown factors are not based on actual data and lead to inefficient/inaccurate repair designs.

The purpose of this task was to develop and optimize on-aircraft processing techniques for several commonly used epoxy-based composite prepreg systems and calculate design allowables for the prepreg materials using on-aircraft processing techniques. The evaluated composite systems were:

- Hexcel AS4/3501-6
- Hexcel IM7/8552
- FiberCote T-700/E765
- Cytec IM7/977-3
- Specialty Materials Boron/5521

At the conclusion of this task, vacuum processing techniques were developed for T-700/E765, IM7/977-3 and Boron/5521. Three batches of mechanical test data were used to generate B-basis allowables for T-700/E765. No usable vacuum processes were developed for AS4/3501-6 or IM7/8552. All of the composite processing methods, test methods, and test data for this program are reported in AFRL-ML-WP-TR-2006-4023.[23]

4 TASK #3: DOCUMENTATION/DISSEMINATION OF GENERAL COMPOSITE REPAIR GUIDELINES

The purpose of this task was to provide significant input to rewrite of the Materials and Processes (M&P) section of the Composite Repair for Metallic Structure (CRMS) Manual. This manual is being updated as part of the Bonded Repair Capability Enhancements program under a task entitled "Updated Bonded Repair Guidelines." Very little work was accomplished on this task by UDRI since the AFRL/MLSA project engineer did not provide the expected workload. A meeting was held at the US Air Force Academy in Colorado Springs, CO, in July 2003, to outline the bulk of the M&P section. However, writing responsibilities and a draft copy of the M&P section were not provided to UDRI by the end of this program.

REFERENCES

[1] R.J. Kuhbander, J.J. Mazza, and J.B. Avram, "Grit Blast/Silane (GBS) Aluminum Surface Preparation for Structural Adhesive Bonding," WL-TR-94-4111, 1999.

[2] S.S. Saliba, "The Surface Preparation of Aluminum Alloys Using the Phosphoric Acid Containment System for Repair," WL-TR-94-4112, 1994.

[3] ASTM D 3933, "Standard Guide for Preparation of Aluminum Surfaces for Structural Adhesives Bonding (Phosphoric Acid Anodizing)." Annual Book of ASTM Standards, Volume 15.06: Adhesives, 1997.

[4] D.B. McCray, "NONMETALS TEST AND EVALUATION Delivery Order 0007: The Development of On-Aircraft Surface Preparations using Sol-Gel Coatings for Adhesive Bonding Aluminum Alloys." AFRL-ML-WP-TR-2002-4131, May 2001.

[5] K.Y. Blohowiak, et.al., "Repair Techniques Using Sol-Gel Surface Preparations," The Second Joint NASA/FAA/DoD Conference on Aging Aircraft, NASA/CP-1999-208982/Part 1, 1999.

[6] W.D. Bascom, "Primers and Coupling Agents," Engineered Materials Handbook Volume 3: Adhesives and Sealants, C. A. Dostal, editor, ASM International, Materials Park, Ohio, 1990, page 254.

[7] D.B. McCray, "The Development of On-Aircraft Surface Preparations Utilizing Sol-Gel Coatings for Adhesive Bonding of Aluminum Alloys," AFRL-ML-WP-TR-2002-4131, May 2002, page 36.

[8] D.B. McCray and J.J. Mazza, "Optimization of Sol-Gel Surface Preparations for Repair Bonding of Aluminum Alloys," Science of Advanced Materials and Process Engineering Series, Volume 45, Society for the Advancement of Materials and Process Engineering, May 2000, page 55.

[9] D.B. McCray and J.J. Mazza, "Optimization of Sol-Gel Surface Preparations for Repair Bonding of Aluminum Alloys," Science of Advanced Materials and Process Engineering Series, Volume 45, Society for the Advancement of Materials and Process Engineering, May 2000, Page 47.

[10] ASTM D 1002, "Standard Test Method for Apparent Shear Strength of Single-Lap-Joint Adhesively Bonded Metal Specimens by Tension Loading (Metal-to-Metal)," Annual Book of ASTM Standards, Volume 15.06: Adhesives, 1997.

[11] ASTM D 3167, "Standard Test Method for Floating Roller Peel Resistance of Adhesives," Annual Book of ASTM Standards, Volume 15.06: Adhesives, 1997.

[12] ASTM D 3762, "Standard Test Method for Adhesive-Bonded Surface Durability of Aluminum (Wedge Test)," Annual Book of ASTM Standards, Volume 15.06: Adhesives, 1997.

[13] D.B. McCray, J.A. Smith, and B.A. Bolan, "The Evaluation of Alternate Positive Pressure Cure Cycles for 250°F-Curing Epoxy Film Adhesives," AFRL-ML-WP-TR-2006-4009 January 2005, Table 2.

[14] D.B. McCray, "Characterization of EA 9696," UDR-TR-2000-00046, March 2000, Figure 4, page 4.

[15] D.B. McCray and J.A. Smith, "The Evaluation of BR 6747-1 and BR 6757-1 Waterborne Adhesive Bond Primers for Aircraft Bonding Applications," AFRL/MLS 00-85, October 2000, page 7.

[16] AC-130 sol-gel, Advanced Chemistry and Technology, Garden Grove, CA, 92841.

[17] J.J. Mazza, G.B. Gaskin, W.S. De Piero, and K.Y. Blohowiak, “Sol-Gel Technology for Low-VOC, Nonchromated Adhesive Bonding Applications,” AFRL-ML-WP-TR-2004-4063, April 2004, pages 65-70.

[18] D.B. McCray, “The Evaluation of Ambient-Temperature Processes for Repair Bonding of Aluminum Alloys,” AFRL-ML-WP-TR-2002-4043, January 2002.

[19] Hysol EA 9320NA Product Data Sheet, Hysol Aerospace Products, Pittsburg, CA 94565, page 2, January 1990.

[20] D.B. McCray, “Characterization of EA 9696,” UDR-TR-2000-00046, Figure 3, page 3.

[21] MIL-HDBK 337, Section 5.3.2.2 “Pasa-Jell 105 Method (Optional),” page 5-20, December 1977.

[22] J.J. Mazza, G.B. Gaskin, W.S. DePiero, and K.Y. Blohowiak, “Sol-Gel Technology for Low-VOC, Nonchromated Adhesive Bonding Applications,” AFRL-ML-WP-TR-2004-4063, April 2004.

[23] B.P. Milligan, D.B. McCray, and P.K. Childers, "The Evaluation of Epoxy-Based Matrix Composite Systems for On-Aircraft Composite Repairs," AFRL-ML-WP-TR-2006-4023, January 2006.